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TEXTILE RESEARCH JOURNAL

VOL. XVII, No. 2 February, 1947

Radiant-Energy Reflectance of Men's-Wear Colors

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SO-CALLED cool colors for men's-wear tropicals have been a point of discussion for some time. Tropical suitings should not, for practical reasons, be too light in color and must be cool for summer wear. In addition to the construction, the weight, the porosity, and other characteristics which would affect the coolness of a fabric, it is believed that the ability of the material to reflect the heat rays of the sun is a contributing factor in a cool fabric. It is presumed that most men's-wear tropical suitings are worn part of the time out-of-doors.

An investigation of the radiant-energy reflectance characteristics of men's-wear colors applied on worsted fabrics was made and the determination of cooler colors was considered.

Résumé of Pertinent Findings

A study of data concerning the distribution of solar energy has shown that the major part of the radiant energy from the sun is almost equally divided in the visible and near infrared regions.

An investigation in this Laboratory of the radiant-energy reflectances of men's-wear colors on worsted fabrics has shown that in general light colors are superior for summer wear. Differences in radiant-energy reflectance between colors which are visually similar were found to be the result of different infrared reflectances; consequently, the factor of infrared reflectance is important when comparison of apparently similar colors is made.

The advantage of high reflectance of extremely light colors is lessened by increased transmittance which is dependent on the color of the fabric as well as on the type of fabric.

Solar Energy

When reference is made to "solar energy" or "radiant energy," the term is used to encompass a broad spectrum including the infrared, the visible, and the ultraviolet regions. Most of the energy from the sun is about equally divided between the visible and the infrared regions, with a small fraction in the ultraviolet.

Professor Moon [4] of the Massachusetts Institute of Technology states in his summary of data of other investigators that with respect to the spectral distribution of solar energy at the earth's surface the peak of the energy is in the blue-green, falling off rapidly to near zero in the middle ultraviolet region and dropping gradually towards the infrared to practically zero in the far infrared, around 2,300 mμ.

Figure 1 represents the solar irradiation at sea level with the sun at zenith, other conditions being the normal and the most characteristic of various places on the earth's surface. The peak of energy

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will shift to some degree as conditions change and the character of the solar energy reaching the earth's surface will be influenced by such factors as position of the sun, the amount of dust, water vapor, and gas molecules in the atmosphere, the absorption of radiant energy by oxygen, ozone, carbon dioxide, etc., but the curve in Figure 1 may be considered to be most characteristic of the distribution of the sun's energy.

H. Laurens [3], Professor of Physiology at Tulane University of Louisiana, School of Medicine, shows that of the total radiation reaching the earth the percentage of ultraviolet is between 1 percent and 5 percent; of the visible, between 41 percent and 45 percent; and of the infrared, between 52 percent and 60 percent. From this, it may be concluded that there is almost as much energy in the visible as in the entire infrared region. Laurens, referring to a comparison of midday summer and winter sun, states that the former is only 10 percent stronger in the infrared than the latter. However, this reference is extended to include a comparison of the visible and ultraviolet regions. In these regions he notes that the summer midday sun is 45 percent stronger in the red, 90 percent stronger in the green, 250 percent stronger in the blue-violet, and 1,000 percent stronger in the ultraviolet than winter midday sun. It is obvious, therefore, that the increase in the heat of the sun's rays in summer is largely attributable to the increase in energy in the visible part of the radiant spectrum.

Professor Laurens, who has compiled the results of extensive work carried on by him and other investigators in regard to the physiological effect of radiant energy, refers to luminous and infrared energy as "radiant heat" and states that irradiation experiments on the skin have shown that luminous rays as well as the infrared warm the tissues.

Opinions regarding the relative penetration of visible and infrared rays into the skin differ, some investigators believing the infrared to penetrate farther, others believing the visible to have greater penetrating power. The investigators referred to by Laurens obtained results from experiments showing that luminous and near infrared energy penetrate farther into the skin than far infrared, warming subcutaneous tissues rather than the outer layers wherein the far infrared is absorbed. The heat produced by the absorption of visible energy is carried away from the deep tissues by the blood stream, whereas the heat produced in the surface of the skin by infrared is only slowly carried away by the blood and by the air.

Dr. Watkins [7], associate in medicine at Harvard Medical School and chief of physical medicine at the Massachusetts General Hospital, refers to studies of skin temperature changes with respect to heat therapy which have shown that penetrating infrared radiation (shorter wave lengths) is more efficient than the nonpenetrating type or than other sources of heat whose transport is largely effected by conduction.

Spectrophotometric reflectance curves of skin show the least reflection of radiant energy in the visible from 500 to 600 mμ, the reflectance increasing toward the ultraviolet and toward the infrared. The energy which is not reflected must be absorbed either in the surface of the skin, or, if it is transmitted to the inner layers, in the subcutaneous tissues. It therefore follows that the luminous energy is absorbed in the skin to a greater degree than is the ultraviolet or the infrared.

In view of the scientific data presented by various investigators, the consideration of visible energy is as important as the consideration of infrared energy in determining the heat reflection of fabrics. Both visible and infrared energy exist in approximately the same percentages of total energy from the sun. Constant irradiation by visible energy which is absorbed in the inner layers of the skin would result in an increase in the temperature of subcutaneous body tissue and irradiation by infrared would effect an increase in surface-skin temperature. However, since both body and skin temperatures are so closely linked, one affecting the other, it may be said that any incident heat-producing energy will influence

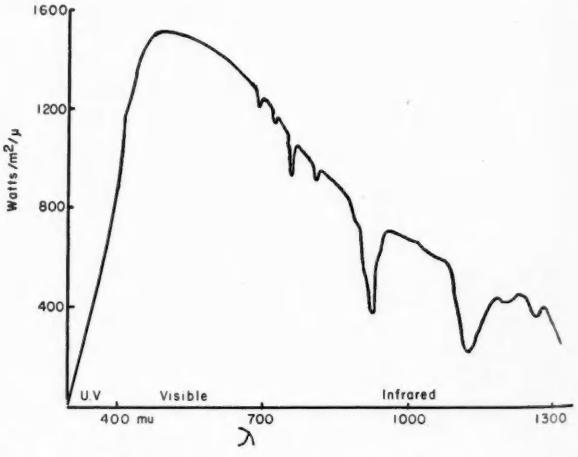

FIG. 1. *Solar irradiation at sea level, sun at zenith.*

TABLE I. MEN'S-WEAR COLORS: MUNSELL NOTATIONS, VISIBLE-ENERGY REFLECTANCE, INFRARED-ENERGY REFLECTANCE, RADIANT-ENERGY REFLECTANCE

Color	Munsell notation	Visible refl. (%)	Infrared refl. (%)	Radiant-Energy refl. (%)	Color	Munsell notation	Visible refl. (%)	Infrared refl. (%)	Radiant-Energy refl. (%)
1	N1	2	12	7	35	5PB2/3	7	50	29
2	5YR1.5/1.5	4	32	18	36	5PB3/2	10	46	28
3	5YR2/2	5	33	19	37	2.5PB3/2	10	48	29
4	2.5YR2/2	5	41	23	38	7.5B3/2	11	43	27
5	5YR3−/1.5	6	37	22	39	5PB3/3	11	60	36
6	10YR2/2	8	48	28	40	5PB4/2	13	51	32
7	5YR3+/1.5	9	35	22	41	6.5PB4/2	14	54	34
8	5YR3/2	9	50	30	42	2.5B5/1	24	63	44
9	10YR4/1.5	12	56	34	43	5BG6/1	32	69	51
10	4YR4/2	13	55	34	44	2.5B6/2	34	64	49
11	5YR4/1	13	59	36	45	5Y2/2	5	41	23
12	5YR4/2	14	58	36	46	7.5GY3/2	8	48	28
13	7.5YR4/2	15	65	40	47	2.5G4/1	12	50	31
14	10YR5−/2	17	50	34	48	4G4/1	13	51	32
15	10YR4/1.5	17	52	35	49	2.5B4/2	14	49	32
16	1Y4/1.5	18	64	41	50	10BG4/2	17	59	38
17	10YR5/4	19	64	42	51	2.5GY5/4	19	60	40
18	5YR6/2	20	50	35	52	2.5PB3/1	9	38	24
19	5YR5/1.5	20	62	41	53	5PB3/1	11	53	32
20	7.5YR5/4	21	62	42	54	N4.5	18	61	40
21	10YR5+/2	22	64	43	55	2.5PB5/2	20	52	36
22	7.5YR5/2	22	71	47	56	N5+	22	61	42
23	2.5Y5/2	24	62	43	57	N5	22	63	43
24	1.5Y5.5/3	26	58	42	58	N5.5−	24	65	45
25	10YR5.5/3	27	65	46	59	10PB3/8	14	62	38
26	1YR5.5+/3	30	70	50	60	5R4/14	21	72	47
27	7.5PB2/1	3	21	12	61	5Y6/6	30	68	49
28	4PB1.5/1.5	4	25	15	62	2.5Y6/4	35	74	55
29	7.5PB2/2	4	35	20	63	5Y7−/4	43	74	59
30	2.5PB2/2	5	30	18	64	5Y7+/4	44	70	57
31	6PB2/3	5	33	19	65	2.5Y7/5	44	75	60
32	5PB2/2	5	35	20	66	2.5Y7.5/4	46	69	58
33	4PB2/2	7	36	22	67	5Y9/2	65	77	71
34	5PB2/4	7	38	23					

body comfort. Since both visible and infrared energy are contributing factors in the heating of the body, the reflection of both by a fabric should be considered.

In the subsequent report of the reflectance characteristics of men's-wear colors, the term "radiant energy" will be used to denote the visible and near-infrared band from 400 to 1,000 mμ, the region in which most of the solar energy occurs and the region in which the reflectance measurements were made.

Previous Investigators

Although much work has been reported on measurements of solar radiation, little has been published on reflection of radiant energy from textile materials. One investigator, J. Gregory [2], made radiant-heat transmission, absorption, and reflection measurements on a number of types of fabrics. Observations on the effect of the color of a fabric on the absorption of radiation were made by W. H. Rees and L. W. Ogden [5]. The source of radiation used was a high-intensity carbon arc, which closely assimilates sunlight in spectral characteristics; thermometers were used to determine rise in temperature due to absorption of incident energy. The results obtained showed that definite variations were effected in the absorption of energy by a fabric, according to the shade of the fabric.

Study of the reflection from textiles of the infrared fraction of solar energy has been carried on by several investigators. P. W. Cunliffe [1] conducted infrared reflection measurements on dyed materials by means of photography. A paper by E. Stearns [6] refers to infrared reflection characteristics of certain materials in regard to camouflage.

Radiant-Energy Reflectance of Men's-Wear Worsted

In the investigation of the radiant-energy reflectance characteristics of men's-wear colors, spectrophotometric measurements were made in the visible and in the near infrared regions on samples of worsted fabric representing many colors used on men's wear.

The instrument employed was a General Electric Recording Photoelectric Spectrophotometer with a 10-millimicron wave length band. Reflectance measurements were made in the visible region from 400 to 700 mμ, and after required adjustments, in the near infrared to 1,000 mμ. The visible and infrared energy reflectances were obtained from the resulting curve by integrating each region. The average for the entire region, 400 to 1,000 mμ, gave the "radiant-energy" reflectance of the color.

If the character of the reflected solar energy is to be considered, calculations involving the reflectance curve of the color and the solar energy distribution curve can be made in the usual manner. However, since it is merely the reflecting power of the color in the proper spectral region with which this paper is primarily concerned, such procedure has been omitted and reflectance only is referred to throughout.

To present a simple description of the colors measured, the nearest Munsell notations were determined; these are listed in Table I. Since some colors had no counterpart in the Munsell *Book of Color* in the possession of this Laboratory, estimates had to be made as to the nearest intermediate notations. The average visible and infrared reflectance values and the radiant-energy reflectances obtained from the measurements are also listed in Table I. It should be pointed out that the values as presented in the table apply to the characteristics of the color alone and, consequently, in any consideration of the data it should be remembered that these values may be altered by the type of fabric involved.

In comparing the average reflectances of the colors in various color groups it was found, in general, that of the samples measured light colors exhibited greater radiant-energy reflectance than the medium or dark colors. There was, of course, some overlapping, as is indicated in Figure 2.

A critical inspection of the data indicated that those colors which exhibited high reflectance in the visible region generally exhibited good infrared characteristics (Figure 3).

Color Selection

In considering cooler colors for outdoor wear, reference to the visible and infrared fractions of solar energy indicates the necessity of having high reflectance in both regions. However, if a selection is to be made on this basis, some arbitrarily selected value must be set as a minimum. A 35-percent reflectance of radiant energy was chosen because that value seemed to fit satisfactorily into the general arrangement of colors as studied in the trial and it represented an approximate average of the reflectances measured.

As may be noted in Figure 2, all the light colors reflected more than 35 percent of radiant energy and therefore would be classed as cool colors.

In making a selection between two or more colors which appear equally light, comparison of the infrared characteristics will determine the more desirable shade. For example, colors 22 and 18, light tans,

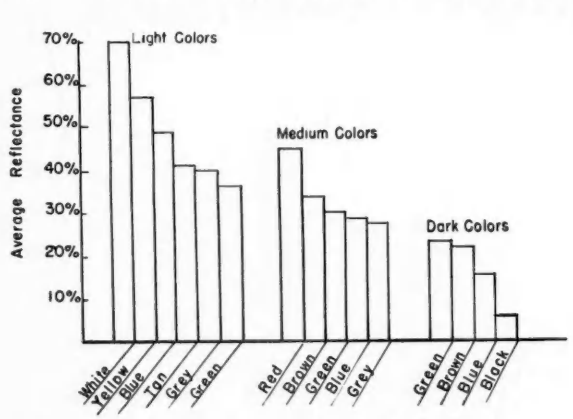

FIG. 2. *Radiant-energy reflectance of men's-wear colors.*

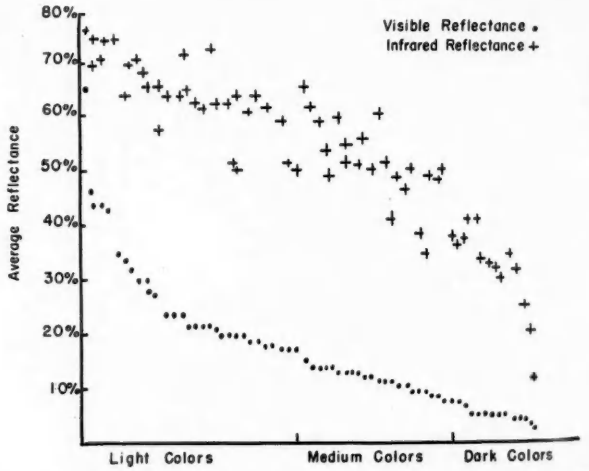

FIG. 3. *Visible and infrared reflectance of men's-wear colors.*

were somewhat alike visually but a comparison of their reflectance curves in Figure 4 shows a considerable difference in the infrared.

Similarly, in the medium browns, colors 8 and 7 appeared to match approximately, but Figure 5 discloses a difference in the infrared region.

In the consideration of dark colors, energy in the visible region will be nearly completely absorbed by the material. Therefore, the problem reduces itself to a study of the characteristics of the dyed material in the infrared, variations of which are shown in Figure 3. In considering, for instance, the dark shades of brown, where there is little difference in visible-energy characteristics the infrared reflectances would indicate that colors 6 and 4 are the best in this group. Likewise, colors 32 and 29 would be best of the dark blues where there is little difference in the visible region.

It is possible for a color to appear darker than another and yet to have higher reflectance of radiant energy. Several examples of this were noted. For instance, color 13, which appeared darker than color 14, has a 40-percent reflectance of radiant energy whereas color 14 has but 34 percent.

Related Factors in Fabric Coolness

Although it is the purpose of this paper to demonstrate the importance of radiant-energy reflectance of men's-wear colors, it is fully realized that a number of other physical factors influence coolness. The importance of air permeability and water-vapor permeability are well recognized. It is also obvious that the radiant energy that is not reflected must either be absorbed by the fabric or transmitted through it.

Transmittance of radiant energy by a fabric is dependent on the construction and finish as well as on the depth of shade. Obviously, a heavily felted fabric will allow less light to pass through the interstices than one of an open construction. At the same time the depth of shade has a direct effect on the transmittance, the darker shade absorbing more of the incident energy and consequently decreasing the amount of energy transmitted.

In the limited investigation of the relative transmittance of men's-wear samples studied in this report, it was found that although white exhibited the greatest reflectance, the white material also transmitted more energy than any of the other colors measured. The white would, therefore, be the coolest color by reason of its reflectance, but lightweight men's-wear fabrics in white would tend to be transparent and hence impractical for general wear. If, to offset this factor of transparency, a heavier or more felted fabric were used, the air permeability of the fabric would be diminished, and the advantage of the high reflectance of the white would be lost. Transmission was found to be reduced considerably in the dyed fabric and it was found that even in the lighter colors in men's-wear transparency was negligible. However, it should be pointed out that the ability of the fabric to absorb energy is generally increased with the depth of shade. Consequently the factor of decreased transmissivity reaches a point in darker colors beyond which it is no longer an advantage, owing to the higher absorption of radiant energy. Therefore, recognizing the influence of both transmittance and absorption as important related factors in fabric coolness, these factors should be considered simultaneously with reflectance when selecting cool colors.

Practical Application

To support the data presented in this paper, a test was conducted in which a number of sport shirts were worn by several individuals. The shirts were made up in colors selected particularly for their

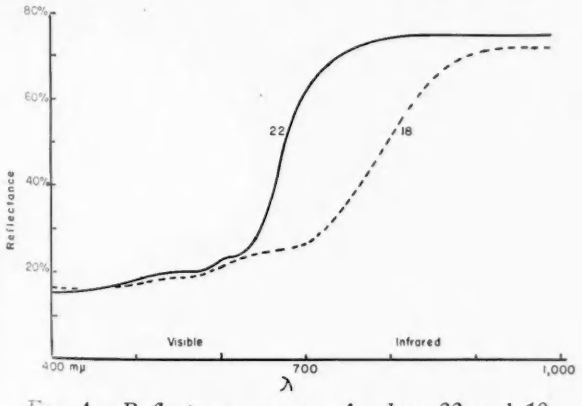

FIG. 4. *Reflectance curves of colors 22 and 18.*

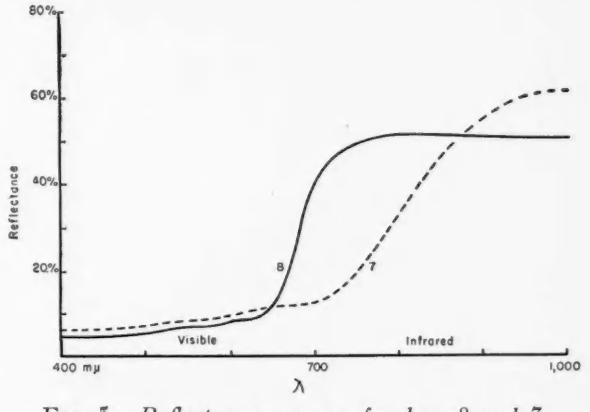

FIG. 5. *Reflectance curves of colors 8 and 7.*

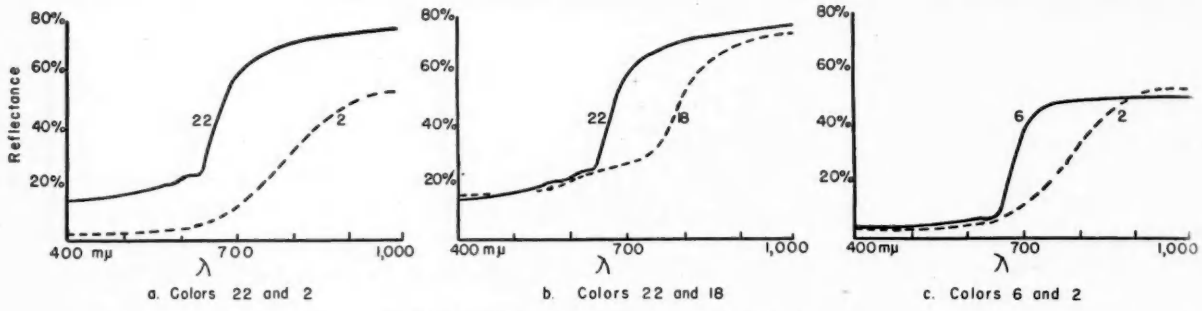

FIG. 6. *Reflectance curves of shirts.*

superior or inferior spectral characteristics. Each participant wore alternately two shirts of different radiant-energy reflectances intermittently for a period of about 3 months in the summertime and was requested to express his opinion as to their comparative coolness.*

The shirts were made of worsted fabric (4.5 ounces per square yard) dyed in four different colors, two tans and two browns, selected from the men's-wear colors studied in this investigation. Color 22 was selected as the best of the tans, with a radiant-energy reflectance of 47 percent, and color 18 as a poorer color, with 35-percent reflectance. The dark browns selected were colors 6, the best in this group with a reflectance of 28 percent, and 2, the poorest of the group, with but 18-percent reflectance. The spectral characteristics of the two tans showed that the greatest difference between them was in the infrared region, the visible reflectance being almost the same. Likewise, the difference between the browns was due, for the most part, to the difference in infrared characteristics.

The curves in Figure 6 represent the reflectance characteristics of the shirts compared in the trial. Figure 6a compares the lighter tan and the darker brown, 22 and 2, respectively; Figure 6b compares the two tans, 22 and 18; and Figure 6c compares the two browns, 6 and 2.

In comparing the tan and brown shirts, 22 and 2, the wearers said that the tan felt noticeably cooler when worn in direct sunlight than the brown. As is shown in Figure 6a, this selection was in favor of the shirt with the higher reflectance characteristics.

In the comparison of the two tan shirts, colors 22 and 18, individuals wearing these colors showed a slight preference for color 22 which was somewhat higher in infrared reflectance characteristics than 18 (Figure 6b), the two shades being similar in the visible region.

A third comparison was made between the two dark shirts, one of which, color 6, exhibited somewhat better infrared characteristics than the other, color 2, but was essentially the same in the visible region. In this case, the wearers stated that no difference could be felt between the two shirts (see Figure 6c).

Conclusion

It may be concluded that radiant-energy reflectance characteristics of a textile material have an appreciable bearing on the coolness of the fabric. This conclusion may be drawn from the reaction of the individuals who wore shirts in the trial and found the shirts exhibiting high reflectance to be substantially cooler than those exhibiting low reflectance.

It would also appear that colors exhibiting high visible reflectance may differ slightly in their coolness because of different infrared characteristics. Apparently, however, this infrared difference must be appreciable before there is a noticeable effect on the coolness of the fabric.

In regard to dark colors, those showing different infrared reflectance do not seem to effect differences great enough to produce a conscious reaction on the part of the wearer.

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* Participants in the trial were not aware that the radiant-energy reflectance of the shirts differed.

Application of Phase Microscopy to Textile Fiber Research

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Considerable attention is being given to applications of a comparatively recent development in the field of microscopy known as "phase microscopy" (also called "phase difference" and "phase contrast microscopy"). An ordinary light microscope is transformed into a phase microscope by placing light-controlling diffraction plates in the objective lens system. By the use of this device the full aperture of the objective lens may be used, thus eliminating the distortion which frequently occurs when the condenser is stopped down and revealing details which are not observable with the ordinary light microscope.

In the United States the Research Department of the Scientific Instrument Division, American Optical Company, has independently developed a phase microscope which is being used by a number of organizations in a variety of fields including biology, medicine, mineralogy, and textiles. Bennett et al. *[1] have described the phase microscope and have discussed the theoretical principles upon which it is based. These theoretical principles are also set forth in an excellent paper by Köhler and Loos, a translation of which appears on page 82 of this issue.*

This article, which illustrates typical applications of the phase microscope in the textile field, is a translation of a manuscript obtained by Mr. Werner von Bergen, Technical Intelligence Investigator in Germany, for the Office of The Quartermaster General, from Dr. H. Reumuth, formerly of the Microscopic Laboratory of I. G. Farbenindustrie at Höchst. The translation is by members of the staff of that laboratory.—Editor

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IT would as yet be premature to attempt a complete evaluation of the phase method of microscopy. A procedure discovered some years ago by Zernike [2] was developed for technical use by the Carl Zeiss Works at Jena from 1939 to 1941. Since 1943 some phase microscope equipment has been available to scientists in a variety of major fields, such as general biology, medicine, and bacteriology [3]. Through the courtesy of the Zeiss Works, the microscopy laboratory of Höchst was supplied at a very early date with experimental apparatus. This made possible the first application of phase microscopy to the technology and chemistry of fibers. In 1941 the initial observations and photomicrographic investigations which were carried on by Dr. K. Michel, B. von Elkesparre, and H. Mehlis, yielded remarkable results. For instance, an effort to use the phase contrast (the Carl Zeiss instrument was called "Phako") method to improve the visibility of the finest structural parts of the wool fiber resulted in the finding of an especially interesting compound arrangement of the subfibrillae in the spindle cells of animal hair which can scarcely be perceived by the ordinary light microscope.

While these experiments were being performed, the suitability of the electron microscope for application in the same field was being investigated both by ourselves and by other scientists working independently, and the first comparison of results was published in 1942 [4]. In that report the essential advantages of the phase method of observation of the fine structures of fibers as compared to both the normal microscope and electron microscope methods were emphasized. In observing, for example, the swelling and dissolving of wool fibrillae, the exact details of the action were not always satisfactorily revealed by the normal microscope. This was true in spite of the fact that the resolving power and the contrast were increased by the use of such devices as oblique illumination, dark-field illumination by

means of a Kardiod condenser, and the use of ultraviolet and infrared photography.

Furthermore, the changes of the cell nucleus in the fibrillae and the tendency of the wool spindle cells to become horny toward the tip while growing could not be ascertained by previously available methods without histological staining, which nearly always caused undesirable effects. To state the problem more clearly, in studying the horny characteristics of the wool fiber it is important to know whether or not the cell nucleus remains in the fibrillae. This question is directly connected with any consideration of damage to wool fibers.

The nucleal-reaction of Feulgen could be used only with the lower or bulbous end of the hair, and not with the upper part. The phase microscope has been instrumental in furnishing rapid solutions to the problems connected with bacterial and enzymic decomposition of wool. Cortical cells decomposed by bacteria are highly reticulated. It was found that the fibrilla, previously considered the basic structure of wool, is actually composed of a reticulated system of so-called subfibrillae. Furthermore, vacuoles of such a nature and location that they could be clearly explained as caves of cell nuclei were observed in some fibrillae (see Figures 1a and 1b). In investigating fibrillae taken from hair near the root, the cell nucleus itself, or at least its rudiments, could be seen in some cases. This result could not be obtained with the electron microscope in spite of much untiring research and the many techniques of sample preparation which were tried. However, the existence of subfibrillae at the utmost tips of the wool fiber fibrillae, which are also called spindle cells, has been shown with the electron microscope both by ourselves and by H. Zahn.

The phase microscopy of thin cross sections (1–3 microns) of wool fibers afforded further valuable observations. Whereas before the advent of the phase microscopical method it was necessary to swell or stain these thin sections, now it is possible to recognize fine detail without resorting to these preliminary treatments. By the new method, outlines of the individual fibrillae could be seen in the cross section and the rudiments of the cell nuclei vacuoles became clearly evident.

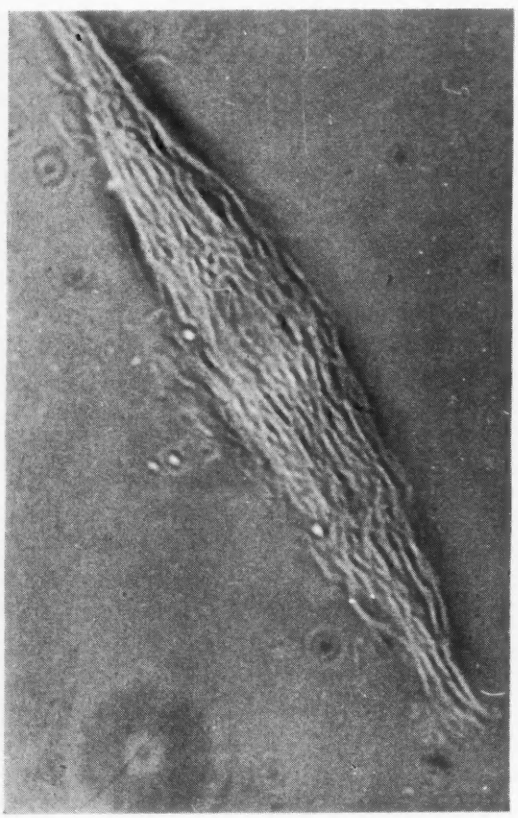


FIG. 1a (*left*)—*Normal.* FIG. 1b (*right*)—*Phase.*

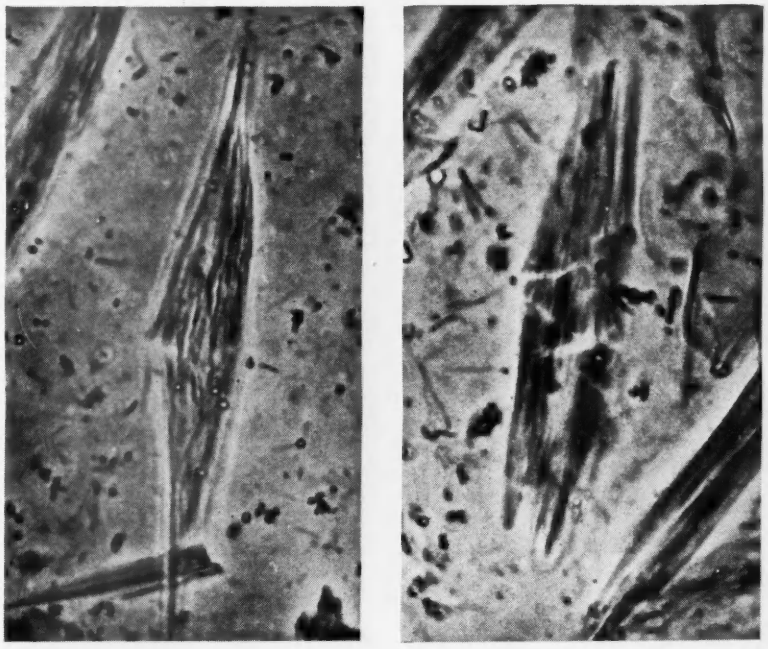


FIG. 2a (*left*). FIG. 2b (*right*).

In January, 1942, new experiments performed at Jena with the phase microscope proved that cortical cells separated bacterially show lines indicating a special type of cross cleavage at an angle which was more or less constant to the longitudinal axis of the fiber (Figures 2a and 2b). Later, Zahn confirmed these observations while investigating wool fibrillae treated with liquid air. Of course, identification of the bacterial and fungic flora in our microscopical preparations has been essentially improved by the use of the phase microscope. In this connection, attention should be called to the work of H. Knoll [5] on the use of the phase microscope in bacteriology.

The phase microscope obviates bacteriological staining in mycological studies on textiles, which in this field is especially objectionable, since not only are the nuclei, spores, and bacteria stained but also the decomposition products of the fibers themselves (see Figures 3, 4, and 5).

In the meantime, we have made completely new discoveries as a result of our attempts to apply to other fibers the phase microscope technique which was first used in the field of wool fiber.

It has already been stated by other workers that in comparison with standard microscopy using bright- and dark-field illumination, phase microscopy is used to advantage only when dealing with fine structures or objects appearing in particularly thin sections. The phase microscope may also be used successfully to ascertain inclusions, turbid portions, samples exhibiting small differences of density, etc.

In textile microscopy the examination of thickening pastes used in printing fabrics is difficult because of their more or less amorphous character. As we have found by recent studies, the depth and brightness of the prints depend in each case upon the state of aggregation and the degree of chemical and, to a lesser extent, of mechanical disintegration of wheat starch, potato starch, tragacanth, or other similar substances.

Furthermore, the penetration into the textile fabric and the general aspect of the finished print depend upon the protective colloid action of the thickening agent. When applying phase microscopy to these thickening pastes extremely important observations have been made. Only a few photomicrographs from a large series are offered as illustrations. Figures 6a and 6b each show a sample of a thickening paste with a wheat starch-tragacanth base to which a resin has been added by emulsification. In Figure 6a, the sample is seen under bright-field oblique illumination.

Figure 6b, on the other hand, which illustrates the application of the phase method, not only presents a better and more contrasting picture but is the only one in which the cellular membrane, partially thrown off after bursting due to chemical decomposition, is shown in true perspective.

It is interesting to note that the large globule of resin which is seen near the lower left side of the photomicrographs is shown in true perspective only by the phase method. From Figure 6b it is clear that this form, which appeared globular in Figure 6a, is actually a particle that has been flattened by the cover glass. Figure 7 shows a thickening paste with a base of esterified cellulose made from technical lignocellulose. While some details of this sample may be seen with oblique illumination (figure not shown), the phase photomicrograph gives a more

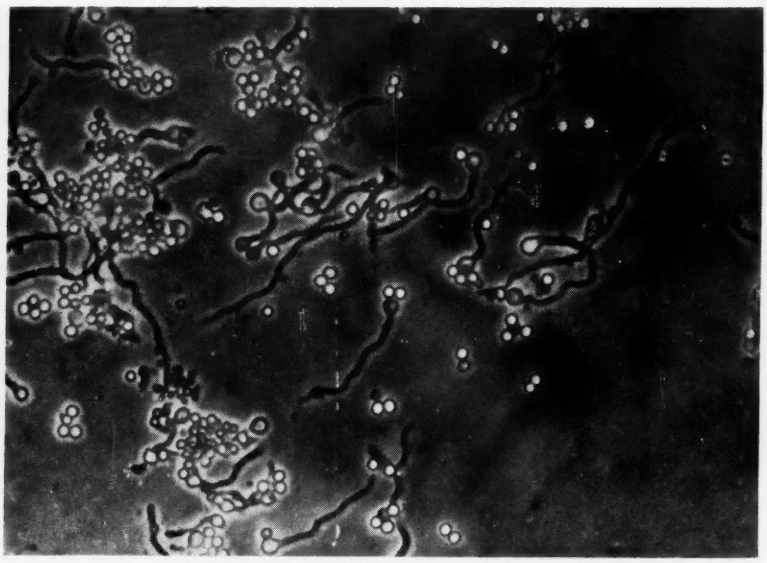


FIG. 3.

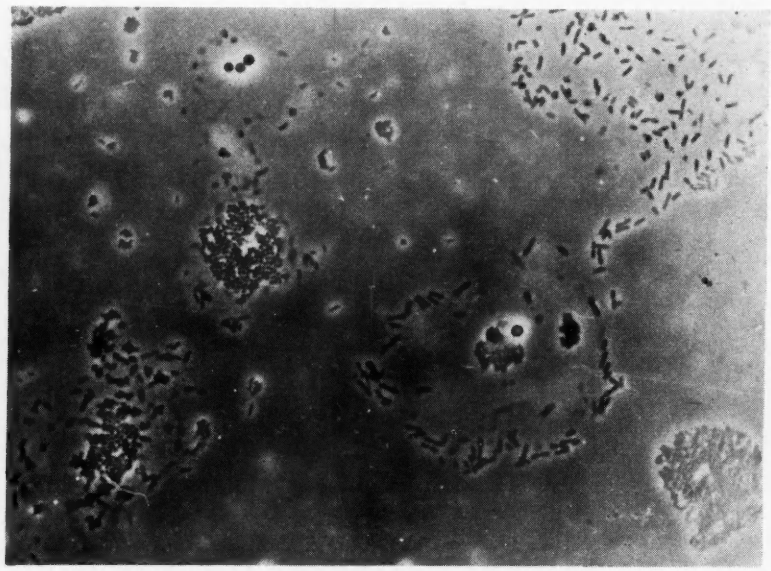


FIG. 4.

complete structural view of the amorphous native fiber particles.

Up to the present, when observing liquid-crystal systems of natural and artificial soaps and lubricants, we have been limited to the use of polarization microscopy. For a thorough examination of such substances, however, the phase technique has been found to be decidedly superior to both the normal and the polarizing methods. The latter is not completely satisfactory, inasmuch as under practical working conditions the microscope must be used with the aperture completely opened for clear observation of the polarization colors obtained between crossed analyzer and polarizer systems, with or without the use of retardation plates. Concentrated solutions of soap form globular crystalline aggregates at room temperature, one shell apparently swimming inside of a larger one. It is possible to view this action through the polarization microscope by constantly rotating the polarizer, but the phase microscope method enables observation of this manifold formation of globular shells within the liquid crystals.

Solid crystals in fine condition or as thin plates are obviously also very favorable subjects for the phase microscope. Figures 8a to 8f are phase photomicrographs of small inorganic crystals found in preparations of wool which had been subjected to natural rotting in the soil for a prolonged period. Both the corrosion of the crystal edges and the gradual dissolving of the crystal faces can clearly be observed. In this case, the chief advantage of phase microscopy—its ability to portray, by difference in shading, slight differences of thickness or elevation—is especially evident.

Our earliest phase microscope observations were made on animal fibers. However, when investigating natural cellulose fibers, such as cotton, ramie, etc., we were unable at first to find any advantages over the known microscopical methods. The apparent failure of the phase microscope technique was naturally caused by the fact that such fibers are not only relatively thick objects but also have a convoluted structure. However, after repeated experiments we discovered phase microscopy to be distinctly advantageous in the study of the structure of swollen fibers. A detailed description of our findings in this instance is offered, since phase microscopy, like every new method, requires much experience in its use before it can be successfully adapted to the investigation of a given type of material. A

cotton fiber is pictured in Figures 9a and 9b, in the former by the usual bright-field illumination and in the latter by the phase method.

While the edges of the fiber are emphasized and the small twisting of the fiber is clearly exhibited by the normal photomicrograph, the protoplasmatic remains in the inner fiber, which are usually not made visible except by histological staining, are distinctly indicated by the phase photomicrograph. It is known that ammoniacal copper oxide solution (Cuoxam) causes the wall layers of cellulose not only to swell but also to dissolve gradually. The spiral lines of these external fiber structures which fail to appear by normal microscopy without oblique illumination or troublesome staining can be easily observed with the phase microscope.

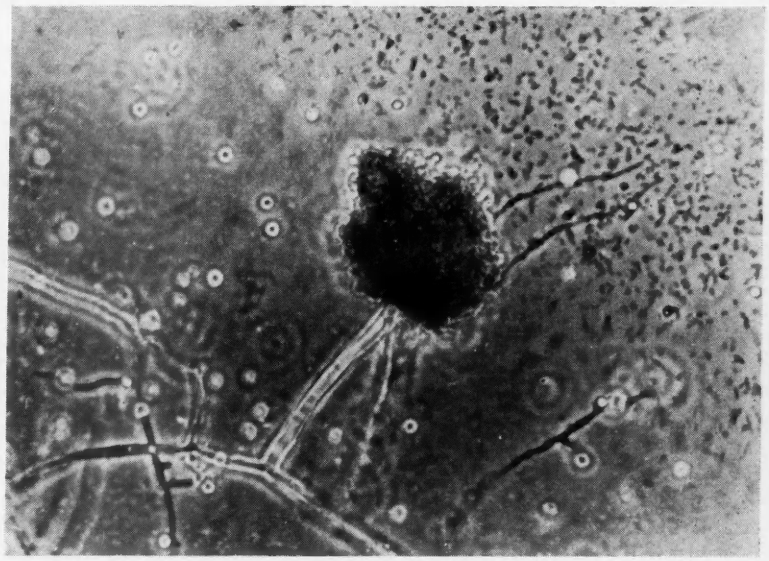


FIG. 5.

These observations were made during the course of our microscopical studies of the silicate residues embedded in fibers by repeated washings with detergents containing this compound. It is well known by students of laundering methods that such materials as cotton that are frequently subjected to the effects

[*Continued on page 78*]

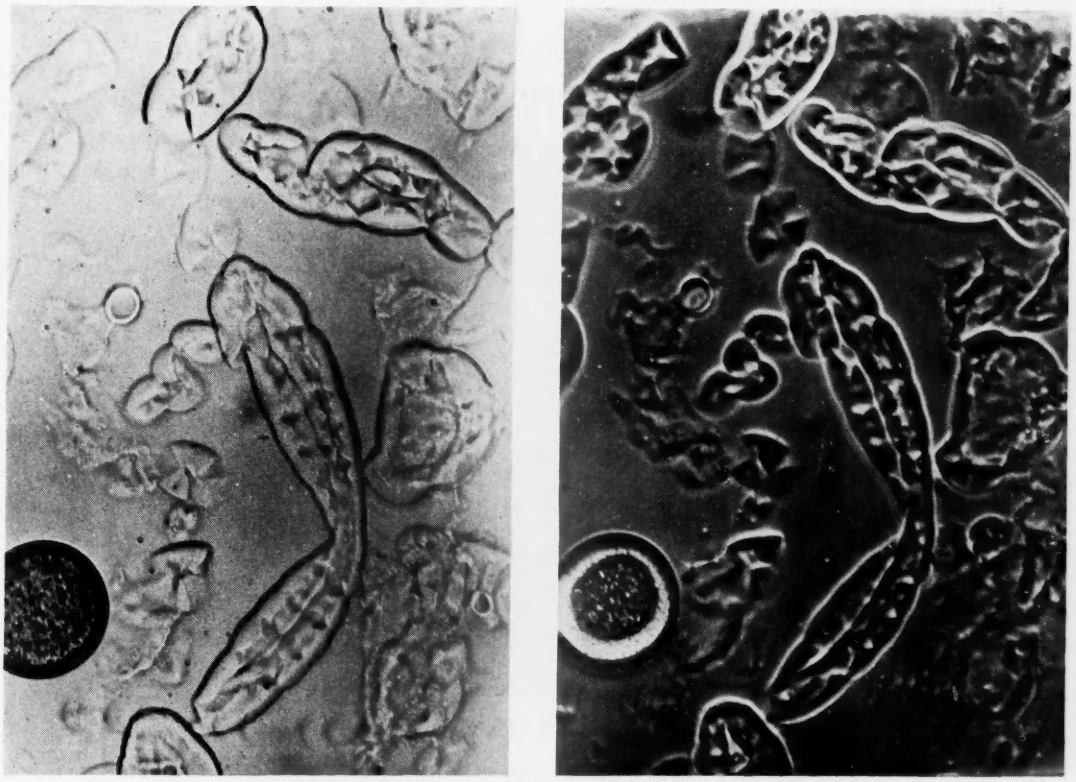


FIG. 6a (*left*)—*Normal.* FIG. 6b (*right*)—*Phase.*

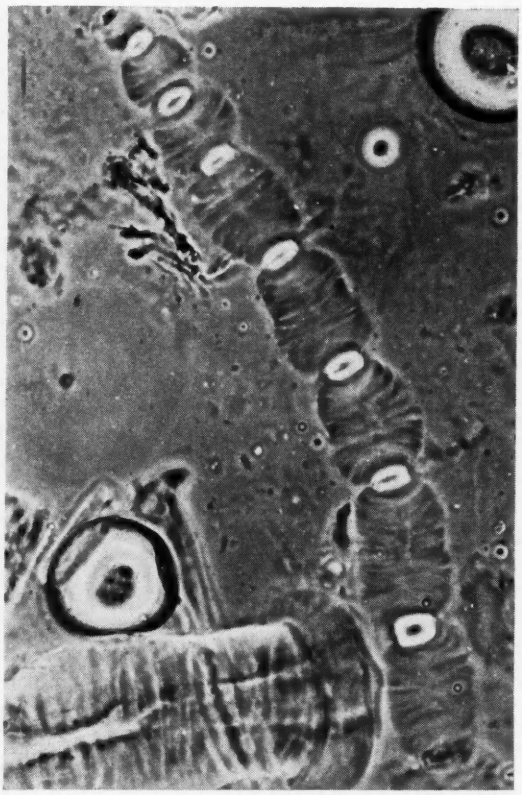


FIG. 7.

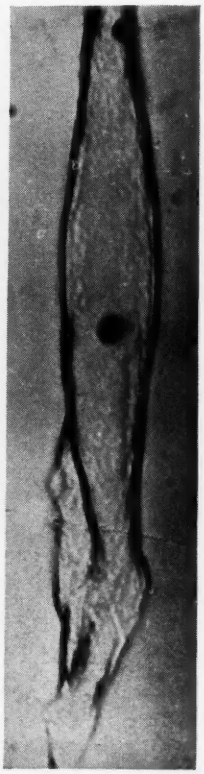
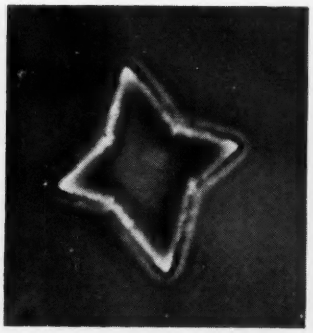


FIG. 9a (*left*). FIG. 9b (*right*).



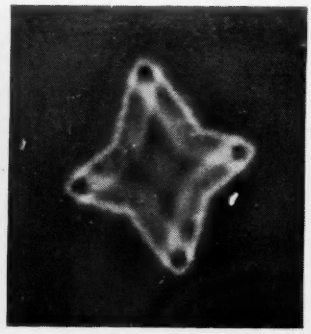
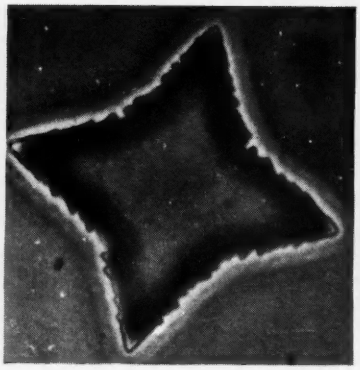
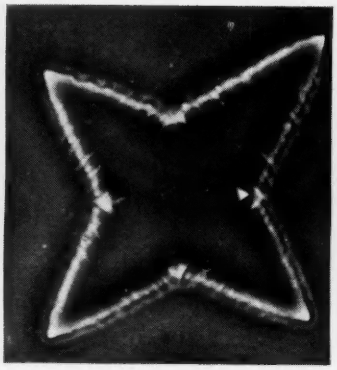


FIG. 8a (*left*).
FIG. 8b (*center*).
FIG. 8c (*right*).





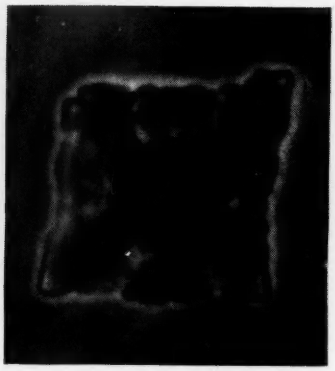


FIG. 8d (*left*).
FIG. 8e (*center*).
FIG. 8f (*right*).

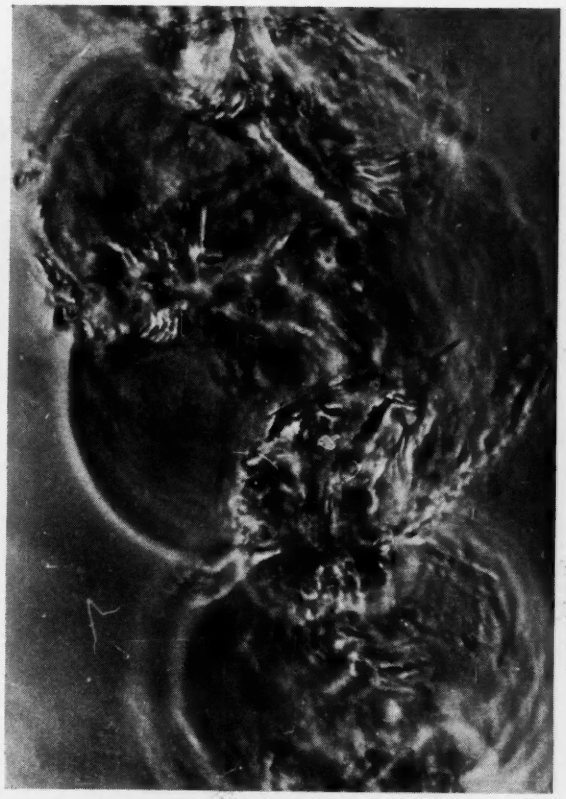
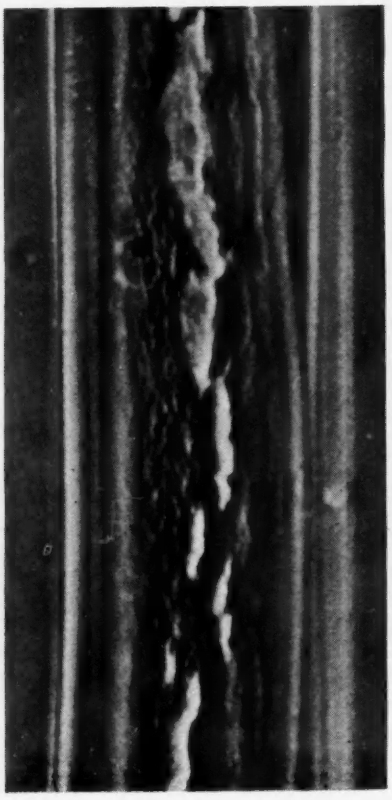


FIG. 10 (*above*).
FIG. 10a (*left*).
FIG. 10b (*right*).

FIG. 10c (*left*).
FIG. 10d (*right*).

FIG. 11a (*left*).
FIG. 11b (*right*).



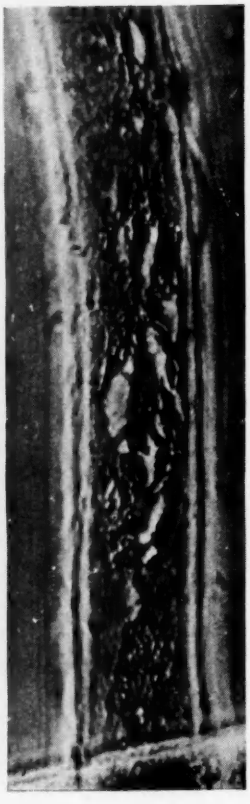
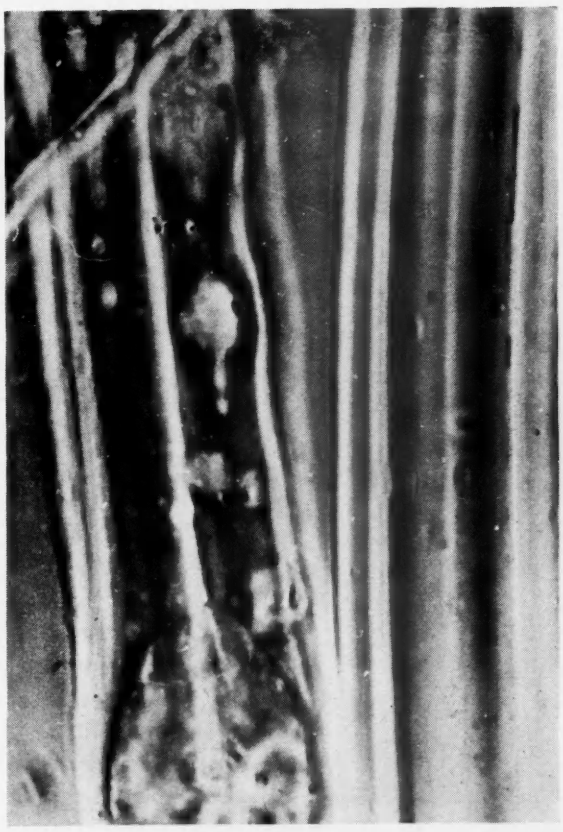


FIG. 12a (*left*).
FIG. 12b (*center*).
FIG. 12c (*right*).

FIG. 13a (*left*).
FIG. 13b (*right*).



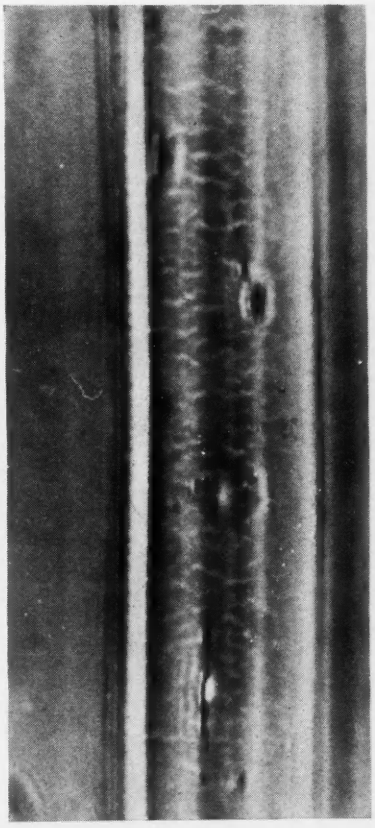
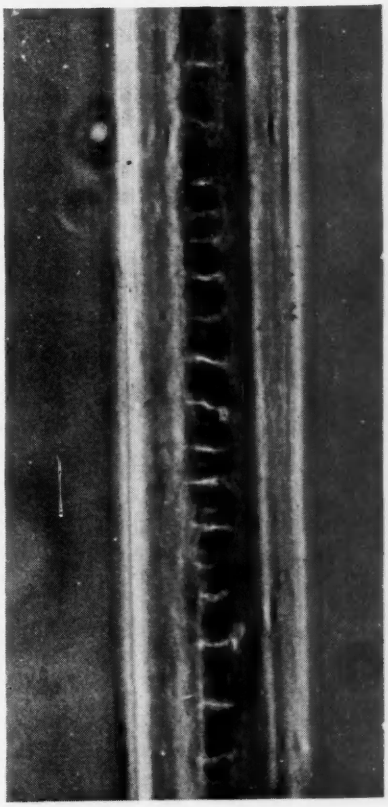


FIG. 14a (*left*).
FIG. 14b (*right*).



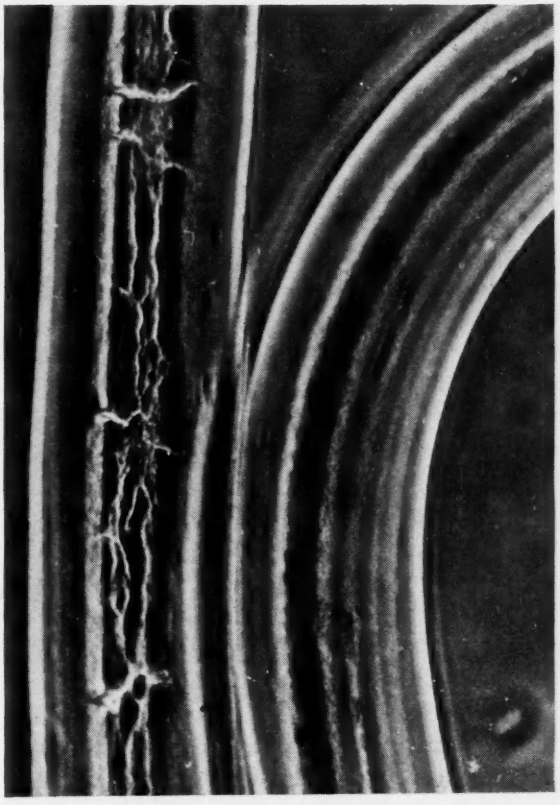


FIG. 15a (*left*). FIG. 15b (*right*).

[*Continued from page 73*]

of washing agents containing silicates may contain traces of mineral deposits. There was no agreement, however, as to the extent to which these minerals had penetrated into the fiber. Whether their presence was superficial or deeply entrenched and whether they were merely in or near the lumen of the fiber or were actually within the micellae of the wall layers of cellulose remained unknown. The phase microscope was particularly well fitted to cope with this situation, since it was necessary to observe small undyed particles embedded within the fiber which have refractive indices very similar to the index of the fiber itself. Figures 10a to 10d illustrate the effectiveness of the instrument. Figure 10a shows a native unwashed cotton fiber considerably swollen by Cuoxam, whereas 10b pictures a fiber of the same origin, similarly swollen, but subjected to 50 launderings with detergents containing silicates. The difference is immediately apparent. The former photomicrograph shows the natural substances of the lumen with the protoplasmic remains already observed in Figure 9b, while Figure 10b indicates the presence of an appreciable number of occlusions; obviously these silicates were not there originally. Owing to their tendency to swell into a globular form and to the spatial winding of the cotton, doubt could arise as to the actual location of the silicates. However, such doubt is dispelled by Figures 10c and 10d, which also present native cotton fiber both before and after 50 launderings. Here the deposition of the silicate is seen to be exclusively within the lumen, but not within the cell wall layers.

The results of these observations suggested similar investigations with respect to synthetic fibers which, apart from occasional technical impurities, are usually considered to be completely homogeneous. The phase microscope, however, reveals that there is no homogeneity of synthetic fibers or of fibers made from regenerated cellulose, regardless of the spinning process employed. Figures 11a and 13a show two kinds of such regenerated cellulose fibers in their natural state just after leaving the mill. The first is a special staple fiber made according to the funnel spinning process, while the second is a commercial viscose staple fiber obtained by precipitation in the Muller bath. The cross section of the funnel-spun fiber is round-to-stlightly-oval, and the longitudinal view of it by the phase method is without the shading which is evident in ordinary microscopy, especially by oblique illumination. Instead, the picture reveals the actual spatial relationship of the fiber, showing the thin marginal sections as lighter and the thick core as darker. Two viscose fibers are shown in Figure 13a, the left strongly intermingled with bubbles, and the right almost without bubbles, deeply furrowed by virtue of its indented cross section, and showing a natural shading proportional to its thickness. Usual bright-field illumination is known to indicate little more than the limiting lines of the single fiber ribs so that bubbles such as those observed in the left-hand fiber of the picture, being objects without much detail, would be represented as black inclusions resembling air bubbles in a mounting medium.

In the course of an extensive series of experiments to determine the effect of the washing process on materials, staple fibers produced from regenerated cellulose were investigated, as cotton fibers had been, by the phase microscope method. Figure 11b shows

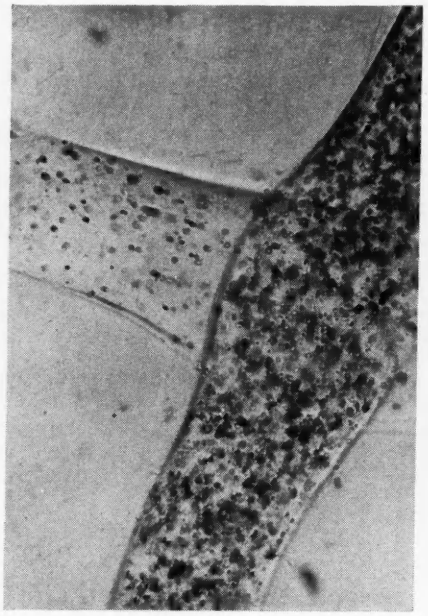
FIG. 16a (*left*). FIG. 16b (*right*).

the staple fiber after ten launderings in an alkaline solution. Here the extent to which it has swollen is not so important as the surface distintegration, which may be explained as the result of mechanical rubbing. The susceptibility of the entire group of textile fibers obtained by the so-called funnel spinning process to become fractured (or splintered) into fibrillae on their surface is well known, but it is especially characteristic of the "Cuprama" type. This action can be retarded, by the application of some after-treatments. Figures 12a, 12b, and 12c represent treated fibers after 50 launderings with detergents containing silicates.

By phase microscopical observation these facts were ascertained immediately without histological manipulation or staining. Here the mechanism of penetration of the colorless silicate is similar to that of the silver precipitation test used by Frey-Wyssling in studies of ramie and other fibers. Not only did this observation answer a practical question, but it allowed the microscopist a further insight into the fine structure of the fiber.

While Figure 13a shows a viscose fiber as it came from the mill, Figure 13b is a phase photomicrograph of the same type of fiber after 50 launderings with water alone. Surprisingly, numerous fine fissures are seen to have been formed across the longitudinal axis, apparently caused by the overstretching or overextending of the wet fibers. Up to the present, discovery of these clefts has been impossible by means of bright-field observation without swelling and prior staining; even with dark-field illumination they have been most difficult to perceive. When similar fibers, however, are subjected to 50 launderings with detergents containing silicates, as shown in Figures 14a and 14b, it is clearly indicated that the compound in the washing agents does become embedded in the fiber core. Its presence there destroys the elasticity of the core, while the skin of the fiber is still capable of elongation without breaking because it is either free of the foreign matter or contains much less of it. It is known that viscose fiber skin has a higher degree of orientation than the core and also differs with respect to dyeing and physical properties. Of the two fibers shown in Figure 14b, the one on the left has been fractured, not only crosswise but, to a great extent, longitudinally. On the other hand, the fiber on the right, though bent, still retains an unbroken, turbid core, probably because of the fact that the elongation to which it was subjected was not so great as that of the other.

In the course of the washing control series, systematic examinations were made to determine the type of silicate responsible for this undesirable deposition. However, the only aspect of our study that is pertinent to this report concerns the methods of microscopy employed to ascertain whether or not a deposition had occurred on the surface in addition to the penetration of the silicates into the core. The issue was settled by a simple phase microscope observation, illustrated in Figures 15a and 15b. In



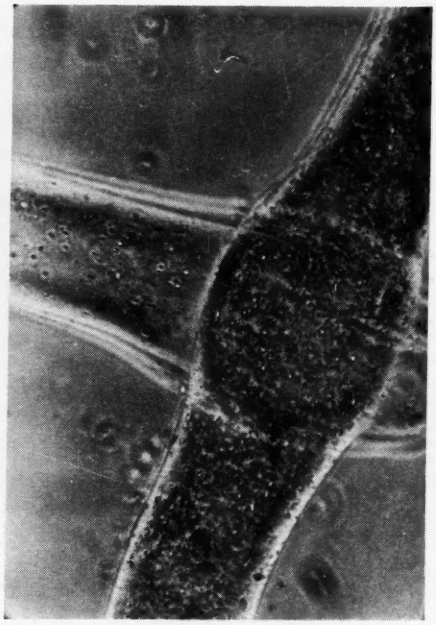


FIG. 17a (*left*). FIG. 17b (*right*).

the former picture is shown a different type of viscose fiber with a strongly granulated core intermingled with silicates. In the outer-wall layer of the cellulose can also be seen a number of bubbles. Figure 15b, on the other hand, clearly indicates the presence of additional rod-like silicate deposits on the surface of the fiber, located in the depths of the ribs. The fibers in Figures 11 to 15 have been clarified by mounting in a 10-percent sodium hydroxide solution and are accordingly in a swollen condition. Clearer views of staple fibers containing silicates are obtained by dissolving them partially in "Cuoxam," as is indicated by Figures 16a and 16b. Here it can be seen that when the wall layer is dissolved the rod-shaped silicates which are outside in the rib valleys crumble off. Then the fiber core containing silicates is dissolved by the action of "Cuoxam," as shown in Figure 16b, leaving the silicates free to swim away with the solvent.

In presenting these photomicrographs we have not intended to exaggerate the advantages of phase microscopy by failing to show corresponding normal light-field views in the best manner possible. On the contrary, when the bright-field diaphragm was substituted for the phase unit the aperture was reduced. Although this procedure revealed an increased amount of detail in the field, it also resulted in the exaggeration of certain defects caused by the diffraction lines and the shadows of foreign substances such as dust on the cover glass or below the slide. One disadvantage of the phase method is that the use of oblique illumination is impossible because the condenser lacks the high numerical aperture of the Abbé condenser. Most of the best photomicrographs have been made with oblique light by substituting the Abbé unit for the phase condenser. All photomicrographs were taken with the same phase objective and a Zeiss trichromo filter. Only in the examination of delicate structures was appreciable success achieved by using an apochromatic objective instead of the phase objective.

It must be stated that there are some limitations to the phase microscope method as compared with normal bright-field microscopy. Mention has already been made of its general failure with respect to observation of very thick objects because its use tends to make small differences in thickness seem immaterial by its inability to indicate their proportional dimensions. This difficulty may be overcome, however, by examining microscopically a part of the object instead of the entire object—for example, a rib of a viscose fiber. Another failure of the phase microscope method is in the observation of crushed cupra staple fibers delustered during the spinning process. Phase microscope pictures of these fibers are most disappointing in comparison with those obtained by normal bright-field illumination, as they show the pigments in poor detail. The phase microscope method is not capable of yielding satisfactory results where such great differences of refractive

index between fiber and delustering agent exist; this is, in fact, beyond the purpose for which the technique was devised. Nevertheless, a study of the phase photomicrograph shown in Figure 17b is essential to an understanding of the bright-field view (Figure 17a), even though the latter is admittedly clearer. Only in the phase photomicrograph are the true dimensional relationships indicated, and only here can it be seen that the thinning of the substance in the fractures brings about a brightening of the designs.

Summary

The progress made by phase microscopy in the field of textile research is of the greatest importance, although the advances may not be fully recognized by those unacquainted with the matter. This technique allows not only the observation of very thin objects, film-like layers, or amorphous masses, but is also of great value when a detailed representation of thick objects is required, which will show the precise arrangement of their high and low or thick and thin layers as well as their actual spatial proportions so that they may be viewed in a topographically correct manner. It is to be assumed that further observations will be made and that new knowledge will be gained following these first important achievements in the field of fiber research.

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The Phase-Contrast Method and Its Applications in Microscopy*

A. Köhler and W. Loos

I

The simplest means of intensifying the contrast of the microscopic image of unstained specimens consists in reducing the aperture of the microscope condenser diaphragm as far as is consistent with the simultaneously decreasing intensity of illumination. The gain in contrast is, to be sure, not very great; in many cases it is even quite inadequate. Moreover, the decrease in the condenser aperture brings about an undesirable loss in resolving power. Another possible manner of increasing the contrast consists in the application of oblique illumination, which under certain circumstances results in obtaining images with a remarkable relief effect; they appear to be plastic and are often quite instructive. However, for various reasons this method has not come into much favor and will therefore merely be mentioned here. Much greater importance is attached to dark-field microscopy than to the two methods mentioned above because of the substantial improvements made by Siedentopf and Zsigmondy [1].

This method is indispensable as an aid to investigation in the fields of colloidal chemistry and bacteriology. Without going into further detail regarding the optical principles involved in this field, it might merely be stated that, in spite of its limitations, as yet the dark-field microscope yields the best contrast intensification of the images of unstained specimens. The contrasts are often reproduced inversely; what would have to appear dark in conformity with the nature of bright-field microscopy here appears bright, and what one would expect to see bright appears dark. Secondly, the contrasts are greatly exaggerated; the differences in intensity in the image do not correspond to the magnitude of the differences in refraction and thickness of the specimen. Where the dark-field method is applied successfully, this phenomenon is of undoubted advantage. This procedure cannot be employed in other fields, however, because the exaggerated contrasts would lead to an entirely false interpretation of what is seen. Furthermore, there are certain characteristics of the specimen, such as differences in thickness, which even the dark-field image cannot show.

In Figure 1 is shown an artificial preparation. A glass slide has been converted by cautious etching into a phase grating † which in the drawing is represented in cross-sectional form. The grating was embedded in Canada balsam under a cover glass. The dark field can show only the sharp edges, because there the light is diffracted [2]. It does not show which are the spaces and which are the slits, nor does it show whether the refractive index of the spaces is greater or less than that of the slits.

Figure 1 shows that by means of the dark-field method essentially only those edges of the structures are represented that have a higher or lower refractive index or have more absorption than the enclosing medium. The phase-contrast method of Zernike [4, 5, 6, 7] avoids these disadvantages. It is especially suitable for the high-contrast presentation of unstained microscopical preparations, and will now be described in greater detail.

Stained and unstained microscopical preparations may be distinguished by the fact that in the former the elements are differentiated by means of their light absorption, and in the latter by means of their light

† The explanation for this term appears on page 84 ff.

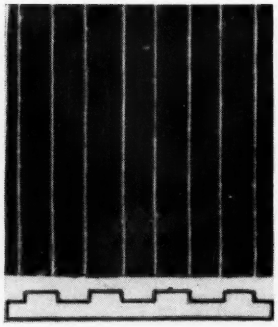


FIG. 1. *Phase grating in the dark field. Underneath, a cross section. Approximately 30:1.*

*From the Micro-Laboratory of the Optical Works of Carl Zeiss, Jena.

Translated from the German paper entitled "Das Phasenkontrastverfahren und seine Anwendungen in der Mikroskopie," from *Die Naturwissenschaften* **29**, 49–61, 1941. (Julius Springer, Berlin.) The manuscript was obtained by Mr. Werner von Bergen, Technical Intelligence Investigator in Germany, for the Office of The Quartermaster General.

refraction. As examples of microscopical preparations, let us first consider simple diffraction gratings in the form of fine slits which are cut at equal intervals, in the first case, in a thin absorbing layer (Figure 3, Ia), and in the second case in a colorless, completely permeable layer (Ib). Let the slits of the gratings be completely filled out by a transparent enclosing medium, which might, for example, even be air. In both instances the light is unaltered as it penetrates the preparation through these slits. As the light goes through the strips it passes, in the first case, the absorption grating with diminished intensity and, in the second, the phase grating with unaltered intensity, but altered phase. If we assume that the layer has a higher index of refraction than the enclosing medium, the light which has passed through the intermediate spaces is retarded in its phase.

The manner of operation of the phase-constrast method cannot be made intelligible without looking more closely into the properties of a wave motion and its representation by means of a vector diagram. Since the representation by means of such a diagram will as a rule not be familiar to the users of the microscope, an explanation is first needed. We shall select as starting point the well-known presentation of wave motion by means of a wave line or sine curve (Figure 2, I). The heavy wave line represents a portion of an advancing wave at a determinate point of time.

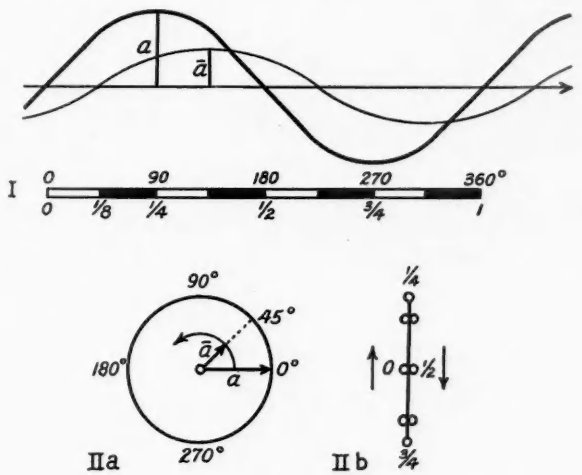


FIG. 2.

I—*Representation of two waves of the same wave length, but different amplitudes* a *and* ā *and different phases.* ā *is 1/8 of an oscillation behind* a. *The numbers under the scale are fractions of a wave length. Above the scale are given in circular degrees the retardations corresponding to the phase differences of 1/4, 1/2, and 1 wave length.*

IIa—*Representation of the same two waves by means of the vector diagram. The length of the vector indicates the amplitude of the oscillation; the angle between two vectors, the phase difference.*

IIb—*An entire oscillation of a particle of a wave* a. *The circles indicate the positions of the particle at the beginning* (o) *of the motion and after the completion of 1/8, 1/4, 3/4, 1/2, etc., of an oscillation.*

It comprises, by analogy with a water wave, a crest and a trough, which together represent a "wave length." Such a wave arises, for example, by reason of the fact that the particles ordinarily move up and down, as the pendulum of a clock moves from right to left. If the wave is advancing in the direction of the arrow, then each succeeding particle begins its motion (its oscillation) somewhat later than the preceding one. If the particle at o (IIb) has gone upward from the position of rest by the distance *a* (¼), then returned into the position of rest (½), and gone downward from this position by the same distance *a* (¾) and arrived at the position of rest for the second time, it has completed an entire oscillation and begins the next one. During this time of a whole oscillation of a particle the wave has advanced by an entire wave length, and the particle at 1, setting out from the position of rest, now begins an oscillation; o and 1 are thenceforward at each moment in the same state of oscillation. We say that they are in the same *oscillation phase,* and this holds in general for any particles whose distance apart is equal to a whole wave length or to a whole number of wave lengths. The greatest deflection *a* represents the width of oscillation or the *amplitude.*

The lighter sine curve (wave line) represents another wave of the same wave length. Its amplitude (ā) is only half as great as *a*. Furthermore, it is not in phase with the first wave. When the particle of the wave *a* in the zero position has completed a whole oscillation and, proceeding upward, has just attained the position of rest, the particle of the wave ā located directly under it must still climb a certain distance; that is, it still needs a certain time in order also to reach the position of rest. This wave motion (ā) is thus retarded with respect to the first, that of *a*, specifically, in the present example, by ⅛ of the duration of a whole oscillation. The time which the wave motion advancing with uniform velocity needs in order to traverse a wave length from o to 1 is of course a whole period of oscillation. We say that the two wave motions have a phase difference, which

can be expressed in fractions of the period of oscillation. If two such wave motions come together, there arises a new wave motion. Its wave length is the same, but its amplitude and phase are in general different from the amplitudes and phases of the two component wave motions. This new wave, the resultant, can be constructed with the aid of Figure 2, I. The representation by means of the above-mentioned vector diagram is, however, simpler and more convient for this purpose. It represents, above all, only the oscillation of a single particle, and specifically we select not an ordinary pendulum oscillation, in which the path of the particle may be regarded as a straight line, but the oscillation of a so-called conical pendulum. Such an oscillation can readily be produced with a pendulum which consists of a weight suspended on a string. In this kind of oscillation the weight passes with uniform velocity through a circle, and the string describes the surface of a cone. The radius of the circle measures the amplitude. Between the paths of a plane and of a conical pendulum of equal amplitude and period of oscillation there exists a very simple relation: the path of the plane pendulum is a diameter of the circular path of the conical one, and the weight of the plane pendulum coincides at each moment with the projection of the weight of the conical pendulum (Figure 2, IIa and IIb).

For our purposes, the conical pendulum has the great advantage that the velocity of the pendulum weight is constant for a whole oscillation, whereas in the case of the plane pendulum it varies between zero (at the two ends of the path) and a maximum (at the two passages through the position of rest).

Figure 2, IIa, shows the vector diagram of the two wave motions represented as sine curves in I. The long arrow represents the amplitude (a) of the one wave, the short arrow the amplitude ($\bar{a}$) of the other. The angle between the two, however, shows the difference in phase. Since the entire circumference, 360°, is traversed during a period of oscillation with uniform velocity, a phase difference of $\frac{1}{8}$ of a period of oscillation appears here as an angle of 360°/8, or 45°, and we have only to add that a rotation in the counterclockwise direction is intended to denote a retardation: the vector diagram then contains everything that we need to know concerning the two wave motions.

In Figure 3, Ia and Ib, the motion of light which has penetrated a grating is represented by such a vector diagram above the slits and spaces. However, not the complete circle but only an arc is shown, which gives the difference in phase.

In the case of the amplitude grating the phases are equal. Hence the vectors lie in the same direction, although their lengths are different; the light passes with unaltered amplitude through the slits, while through the intervening spaces its amplitude is reduced (Ia).

In the case of the phase grating, the behavior is different. The vectors have the same length, since the amplitude does not change, but the phase is different. How this is brought about by the higher refraction number of the spaces will be discussed later; for the present we merely take cognizance of the fact.

FIG. 3 (opposite).

Ia—*Section of an amplitude grating; above it, the light vector over a slit (left) and over a space (right).*

Ib—Section of a phase grating; above it, the light vector over a slit (left) and over a space (right).

Ic—*Diffraction spectrum of an amplitude or phase grating; the maximum 0 resolved into two components, one of which is somewhat displaced upward in order to represent the two separately. P90°, phase plate by which the phase over the maximum 0 is changed by 90°.*

IIa—*The vectors of* Ia *resolved into two components, the larger of which is equal to the vector over the slit.*

IIb—*The vectors of* Ib *likewise resolved.*

IIIa and IIIb—*The larger components alone; underneath, a cut through the plate which produces the light distribution characterized by these vectors.*

IIIc—*The diffraction spectrum (consisting only of the unaltered image of the source of light, the maximum 0) of such a plate.*

IVa—*The smaller component, present only over the spaces of the actual amplitude grating and retarded by 180° in phase; underneath, a cut through an amplitude grating and a phase plate P180° which together produce the light distribution characterized by the smaller vector.*

IVb—*The same representation of the smaller component of the phase grating, and thereunder the section of the corresponding amplitude grating together with phase plate P90°. It is to be noted that* IVa *and* IVb *differ only in the phase plate.*

IVc—*The diffraction spectrum produced by this amplitude grating and by the phase plates. The low intensity of the maxima is indicated by the dark tint.*

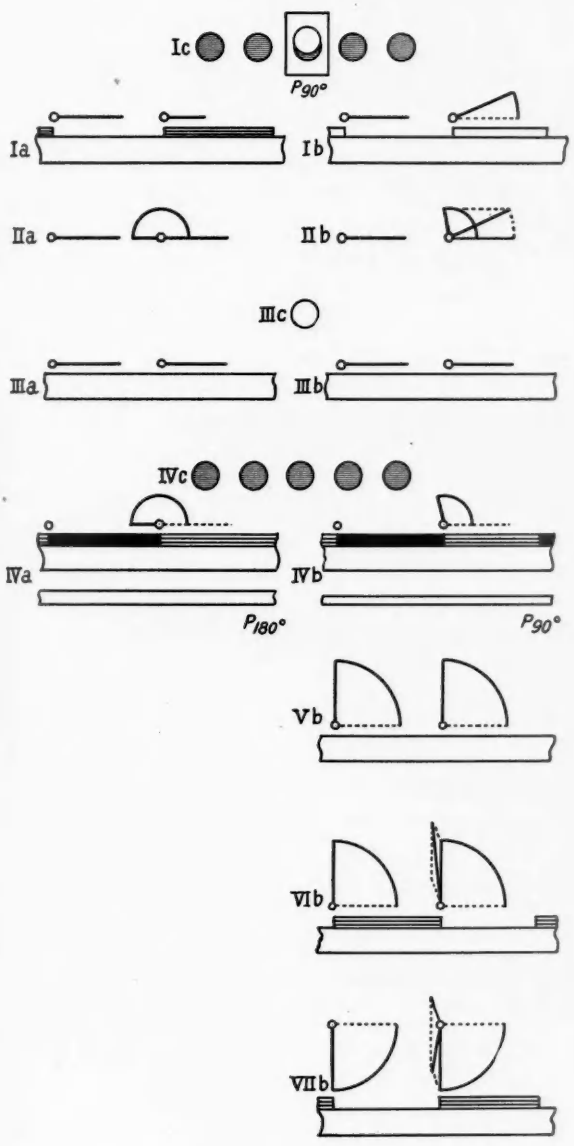
Vb—*The vectors of* IIIb *retarded by 90° as a result of the phase plate P90°* (Ic).

VIb—*The light vectors occurring over the spaces and slits of the phase grating* (Ib) *under the influence of the phase plate P90°, which are the resultants of the vectors represented separately in* IVb *and* Vb. *Underneath, the amplitude grating corresponding to these vectors; negative phase contrast.*

VIIb—*The corresponding representation of the positive phase contrast.*

The retardation will be very slight; hence the angle formed by the directions of the two vectors will be small (in the figure it is very large for the sake of clarity).

Not only does a vector diagram serve to determine the amplitude and phase of a wave motion composed of two different wave motions, but a given wave motion can also be resolved into two, or even more, wave motions. Both operations are made possible by a simple construction which is quite similar to that of the well-known parallelogram of forces. In accordance with Zernike, let us now resolve the light motion over the spaces into two components, one of which exactly agrees in amplitude and phase with the vector over the slits of the two gratings. The vector over the slits is therefore not resolved at all, or the vector itself is, if we wish, one of the components, while the other is zero and has, naturally, neither a determinate amplitude nor phase. This holds, as a matter of course, for both gratings (IIa and IIb). The vector over the spaces behaves differently for the two kinds of gratings. In the case of the phase grating (IIb) a small component appears in addition to the large one which is equal to the vector over the slits. The smaller vector, representing it and the phase angle which it forms with the vector of the large component, more closely approaches 90° as the retardation caused by the spaces of the grating decreases.



In Figure 3, IIIa and IIIb, the large, equal vectors, and IVa and IVb, the small vectors, are once more represented separately.

First, let us consider IIIa and IIIb. The vectors over the spaces and over the slits, being equal in phase and amplitude, indicate that in the plane of the object at the location of the spaces neither absorption nor retardation sets in; hence in this plane no motion of light at all can occur. The two kinds of gratings behave quite similarly with respect to these components. At the place where in Ia, Ib, and IIa and IIb the grating was represented with a plane plate as support, the homogeneous plate, of course, permits the light to pass everywhere with equal phase and amplitude. In IVa and IVb we find the small vectors over the spaces, whereas the vectors over the slits are zero, as already stated above, for all the light passing through is of course represented by the corresponding vectors in IIIa and IIIb. This light distribution would be occasioned by a grating whose spaces and slits exactly corresponded in their width and interval with those of the grating actually present. At the place of the slits, however, absolutely no light should penetrate, and at the position of the spaces an intense absorption of light would have to occur. Furthermore, the phase in IVa would have to be retarded by 180° and in IVb by 90°. Such a grating could be made up of a pure amplitude grating, whose spaces are completely impenetrable and whose slits

are still strongly absorbent. The spaces of this grating would have to lie at the location of the slits of the grating actually present, while the slits would take the position of the spaces actually present. Such a grating is indicated in IVa and IVb underneath the vector diagram. There would also have to be added, however, a perfectly transparent "phase plate," which would retard the phase of the amplitude grating by 180° (IVa, *P*180°) and of the phase grating by 90° (IVb, *P*90°). If we conceive of the image of the plates in IIIa and IIIb and the image of the dark amplitude grating in IVa and IVb as projected upon each other under certain conditions into which we cannot enter more closely at present, the total image would result in either an amplitude grating or a phase grating, depending on whether there was inserted in front of the dark amplitude grating a phase plate *P*180° or *P*90°; this phase plate would be the only difference between the two arrangements.

If it were possible in the case of the amplitude grating to change the phases of the two large vectors over the spaces and slits or the phase of the small vector over the spaces by 90°, the image of the amplitude grating would have to be converted into that of a phase grating, and vice versa. The latter case is the more important; the invisible spaces and slits of the phase grating would then become visible as a result of the different brightness. As Zernike has shown, this is possible. In order to visualize it, we must consider the light distribution in the exit pupil of the objective, which forms the image of the grating. There occurs at that place—for the phase grating as well as the amplitude grating—a Fraunhofer diffraction spectrum which, when illuminated directly with the source of light, is composed of a colorless direct image of the light source, the maximum 0, and the chromatically edged accessory images, the maxima $\pm 1 \pm 2 \ldots \pm m$ (as Figure 3, IVc). This diffraction spectrum must naturally contain as components the diffraction spectra which would be produced by the plane plate (IIIa and IIIb) and the dark amplitude grating with the phase plate (IVa and IVb), each for itself. A plane plate, however, produces no diffraction spectrum; that is, the diffraction spectrum is reduced to the maximum 0, the colorless, nondiffracted image of the light source (IIIc). An amplitude grating with the phase plate, however, produces a Fraunhofer diffraction spectrum which consists of the maxima $0 \pm 1 \pm 2 \ldots \pm m$ (IVc). In order to indicate that this diffraction spectrum is on the whole allergic to light (*lichtschwach*)—even the transparent places should of course let a little light pass—all maxima are shaded (hatched). The two spectra are superimposed in the above-mentioned spectrum produced by the original grating, so that their maxima coincide. In Ic they are drawn separately for the sake of clarity.

We now apply a phase platelet *P*90° (Ic) at the position of the diffraction spectrum, covering only the maximum 0; it imparts to the light passing through it a retardation of 90°, while leaving completely unaltered the light which passes through the maxima $\pm 1 \pm 2 \ldots \pm m$.

Since all the light represented by the vectors (Figure 3, IIIb) passes through the maximum 0, these vectors become altered in phase by 90° and are rotated 90° counterclockwise, falling almost in the direction of the small component (Vb). Over the slits, the vector rotated by 90° remains unaltered (VIb, left), while over the spaces there is further added the small vector (VIb, right). VIb shows also the construction of the resultants from the vectors over the spaces and slits. They have almost the same phase, but are of different length, as in the case of the amplitude grating, and hence the image must represent such a grating. The spaces appear brighter than the slits, as indicated by the shading at the left.

VIIb shows that the opposite occurs when the phase plate *P*90° accelerates the maximum 0 by 90° or retards the maxima $\pm 1 \pm 2 \ldots \pm m$ by 90°, for of course it is only a question of phase difference. The spaces then appear darker, and this contrast will in general be preferred. It is indicated by the hachures at the right.

In connection with further experiments, one of the writers (Loos) made the following interesting observation. Instead of providing the condenser with the normal circular diaphragm (*Ringblende*), the opening of which is imaged exactly upon the phase platelet in the exit pupil of the objective, he used a diaphragm which was opaque in the sections where the normal diaphragm is transparent, and vice versa. Hence it represents the negative of the normal diaphragm. For the sake of brevity, the normal one may be denoted as a "positive" diaphragm and this new one as a "negative" diaphragm.

If, then, the phase-contrast method with the normal positive diaphragm furnishes an image with positive phase contrast, the negative diaphragm furnishes an image with a clearly negative phase contrast (Figure

11, e and f). This is explained on the basis of three facts:

1. In the case of a wide illuminating cone, the image of a phase grating shows a uniformly illuminated surface under the conditions discussed above.

2. The positive diaphragm produces, on the other hand, a certain gradation of brightness corresponding to the structure of the object.

3. According to Abbé, the broad diaphragm presupposed in statement 1 may be conceived as broken up into any desired number of smaller parts, each of which produces a "partial image" (*Teilbild*). Where the adjustment is precise the total image then appears as these partial images coincide, so that, point for point, the *brightnesses* of the individual images are added together. No interference occurs under these conditions.

Hence we may also conceive of the whole diaphragm as resolved into two parts, one of which corresponds to the aperture of the "positive," the other to that of the "negative" diaphragm. The partial images of the object which are produced by each of these diaphragm parts must, according to statement 3, above, add up to the total image produced by the broad diaphragm, and hence, according to statement 1, produce a uniformly illuminated surface.

That is possible, however, only when the brightnesses of the two partial images yield the same sum everywhere at corresponding points; that is, when the images produced by way of a "positive" and a "negative" diaphragm behave as the positives to the associated negatives.

The above general derivation of the method is, however, not quite rigorous. There has been neglected, for example, the circumstance that the phase platelet retards or accelerates by 90° the (rather weak) light of the maximum 0 of the diffraction spectrum, which belongs to the dark amplitude grating (Ic). This need not be considered here, however, where we are merely concerned with an exposition as simple and clear as possible. We have also neglected, of course, in the case of the phase grating, the deviation of the phase angle by 90°. At any rate, the simplified explanation represents the situation correctly, even though quantitatively it is not quite accurate.

One error consists in the fact that the sum of the vectors over the spaces and slits comes out, according to circumstances, larger (VIb) or smaller (VIIb) than it may actually be (according to Ib). Unaccountably, therefore, the intensity of the light permitted to pass by the grating appears on the whole to be increased in the first case, which Zernike denotes as "negative phase contrast," and to be diminished in the second, the case of the "positive phase contrast." Zernike [7] has shown, however, that this difference does not exist. The phase contrast rests only on the fact that the total intensity, while remaining the same, is distributed unevenly over the surface of the image, corresponding to the unequal distribution of the phase retardation over the surface of the object (see also Figure 7). The slight difference in phase between the vectors over the spaces and the vectors over the slits is of no material significance. Most microscopic preparations which contain absorbing elements are by no means pure amplitude gratings, for the absorbing materials as a rule also have refractive indices which differ from those of the surroundings. In addition to shortening, therefore, the corresponding vector almost always reveals a change in phase.

Finally, it might be pointed out that it may be advantageous if the phase platelet gives rise to a certain amount of absorption in addition to producing a change in phase. In this way the contrast may be still further increased. This intensification of contrast through partial absorption of the light contained in the maximum 0 was investigated some years ago by Bratuschek [3]. The way in which this intensification of contrast occurs can likewise be readily observed with the aid of a vector diagram. We think of the light setting out from each maximum of the diffraction spectrum as represented by a vector. We can then form a resultant from the vectors of the maxima $\pm 1 \pm 2 \ldots \pm m$ and with the aid of this resultant and of the vector which represents the light coming from the maximum 0 determine the brightness at each point of the image. Of course the greatest possible contrast will occur when the intensity at individual points drops to zero; that is, when both vectors at these places have the same amplitude and a phase difference at 180°. If, however, the amplitude of the vector (o) is greater than the amplitude of the resultant, an excess of brightness must remain. Since this excess brightness originates exclusively from the maximum 0, it distributes itself uniformly over the entire image like a veil, reducing the contrast. If, however, the phase platelet (*P*90°,

Ic) has a passable absorption capacity, this "veil" can naturally be diminished or eliminated.

In explanation of the difference of phase between the light which has been dispersed through a layer with refractive index (n) and thickness (d) and the light which has passed outside this layer through an enclosing medium of refractive index (n_e) the following may be stated:

The number of wave lengths lying within a plate of thickness (d) when the light penetrates vertically is (Figure 4)

$$x = \frac{d}{\lambda},$$

where λ is the wave length in a medium of refraction number (n). If the wave length *in vacuo* is λ_0, then,

$$\lambda = \frac{\lambda_0}{n}$$

and

$$x = \frac{d \cdot n}{\lambda_0},$$

x is also the number of oscillations which occur within the plate. In the usually thick layer of the enclosing medium the number of wave lengths and oscillations x_e is

$$x_e = \frac{d \cdot n_e}{\lambda_0}.$$

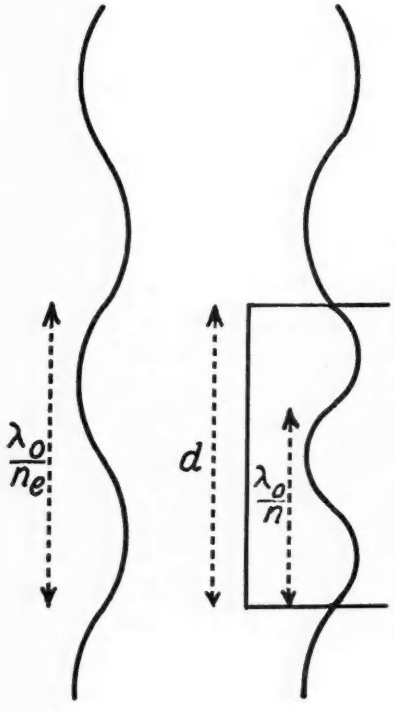


FIG. 4. *Construction of the phase difference between a ray which has passed through a plane parallel plate of thickness* d *and refraction number* n *and a ray which traverses only the enclosing medium of refraction number* n_e. λ_0 *is the wave length in empty space or in a medium having an index of refraction of 1. The difference in travel is 1/2 wave length; the phase difference, 360°/2 or 180°.*

Between the light which has passed through the plate and the light which has passed by it, the difference in the number of oscillations on the same span (d) is

$$x - x_e = \frac{d \cdot n}{\lambda_0} - \frac{d \cdot n_e}{\lambda_0}.$$

If, therefore, in the case of a grating such as that in Figure 2, Ib, the refractive index of the spaces is n, the thickness of the layer d, and the refractive index of the enclosing medium filling the slits is n_e, then the difference in the oscillations is

$$x - x_e = \Delta x = \frac{d}{\lambda_0}(n - n_e),$$

whereas the phase angle, expressed in circular degrees, is

$$\Delta\varphi = \Delta x \cdot 360° = 360° \frac{d}{\lambda_0}(n - n_e).$$

If this phase difference is 90° or ¼ of an oscillation, then

$$\frac{1}{4} = \frac{d}{\lambda_0}(n - n_e)$$

and the required thickness of the plate is

$$d = \frac{\lambda_0}{4(n - n_e)}.$$

In the case of microscopical preparations, however, the relations are in general not so simple as for a grating; there is lacking, particularly, the sharp separation between a maximum 0 and the deflected light. This separation would be present, theoretically, only in case of illumination with strictly parallel light—

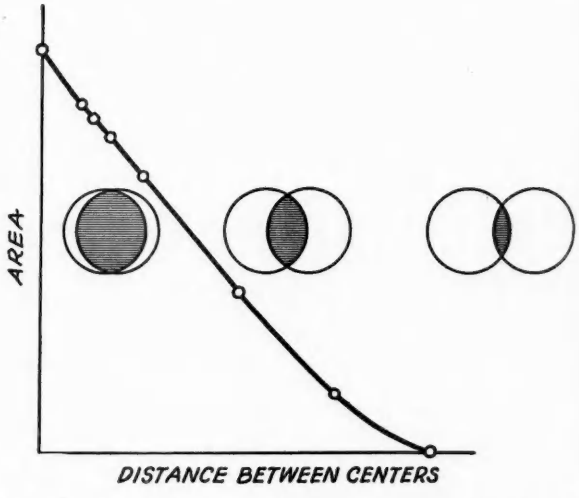


FIG. 5. *Dependence of the overlapping area of two equal circles upon the distance between their centers.*

that is, with a very small light source at a very great distance from the object.

II

In the foregoing discussion, the light source was assumed to be round, and, consequently, its image in the exit pupil of the objective to be a round disc. In order to be independent of the various possible light sources, let us replace them by the diaphragm of the microscope condenser. This diaphragm is uniformly filled with light and imaged at infinity when it is located in the front focal plane of the condenser. The real image of the diaphragm is then reproduced in the substitute exit pupil of the objective.

It is now required that only the light of the maximum 0 be altered in its phase through insertion of the phase platelet. This involves difficulties as long as the maxima are full, round discs. That is to say, the lateral maxima of the diffraction spectrum are truly separated from the maximum 0 only when this maximum itself as well as the grating constants of the microscopic preparation is sufficiently small. Otherwise, as in the case of irregular structures such as are revealed by most of the usual preparations, the lateral maxima partially overlap the maximum 0, so that it is not possible to influence only the light of this maximum in its phase, as is unconditionally required for bringing about the effect. In Figure 5, the dependence of the area covered in common by the maxima 0 and 1 on the distance between the centers of the two circles is represented as a curve. The situation is much more favorable when, instead of giving the maxima the form of a disc, we give them that of a ring (*Kreisring*) (Figure 6). To this end we have

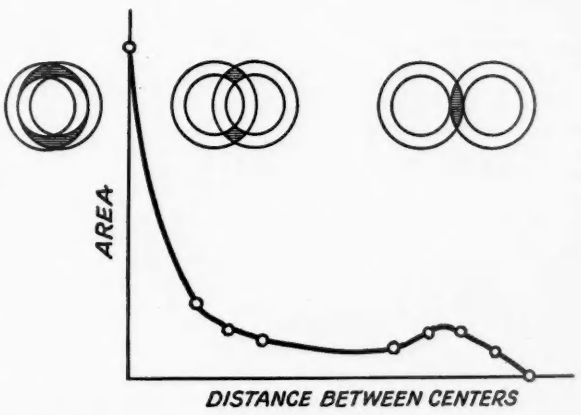


FIG. 6. *Dependence of the overlapping area of two equal rings upon the distance between their centers.*

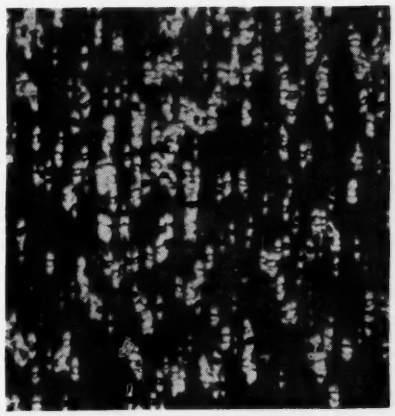


FIG. 7. *Influence of a slit-shaped phase platelet upon the distribution of brightness in the image. Negative phase contrast. 40:1.*

only to replace the iris diaphragm of the condenser by an annular diaphragm. The curve of the dependence between interval and area covered in common is then much steeper—that is, even in the case of a relatively small distance between centers the maxima 0 and 1 are almost completely separated.* Apart from reducing the technical difficulties of execution, there is another reason for which the ring is more favorable for the present purpose than, for example, a figure bounded by straight lines. Naturally the width of the ring and its diameter also play a part. For both, there is an optimum.

In discussing the basis of the method, it has already been stated that the intensity is distributed nonuniformly over the image. This can readily be demonstrated when the condenser diaphragm is designed, for example, in slit form. This phenomenon can be observed especially well on the microscopic image of a preparation which consists mainly of equally large particles with a refractive index similar to that of the surrounding medium (Figure 7). In the case of negative contrast each particle has bright tails on opposite sides, while the tails are seen to be dark by positive contrast. The direction of these tails

*It goes without saying that instead of a single ring, several small concentric rings may be used as a diaphragm. The phase platelet must then also, correspondingly, consist of several rings. In this way we can, under favorable circumstances, obtain greater brightness of the image without essentially impairing the separation between the maximum 0 and the derived maxima. Moreover, there exists the possibility of designing such multiple diaphragms as equivalent "positive" and "negative" diaphragms (see figure herewith), so as to secure with the same phase platelet either positive or negative phase contrast (*cf.* Figure 11, e, f).

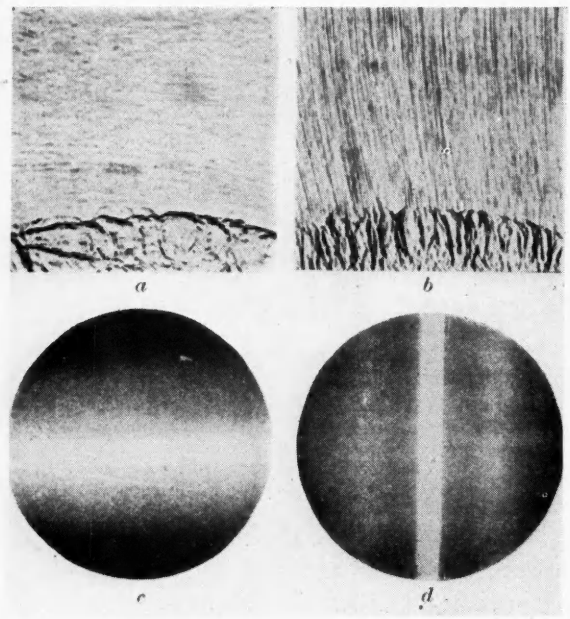


FIG. 8. *Influence of a slit-shaped phase platelet upon the image. (Thin cut through a tooth.)* a—*Dentine fibrils perpendicular to the slit;* b—*fibrils parallel to the slit;* c *and* d—*the diffraction images belonging to* a *and* b, *in the exit pupil of the objective. 40:1.*

is at right angles to the direction of the likewise slit-form diffraction maxima. The brightness (or darkness) of the tails is greatest in the neighborhood of the particles and soon decreases at a short distance therefrom. This phenomenon, which is of importance as proof for the correctness of the theory of the phase-contrast method, could be overlooked, if necessary, as an esthetic defect. However, the slit form of the condenser diaphragm brings about an azimuth error in other preparations which in practice may lead to disastrously false conclusions in judgment of the image. It may happen that elements of a preparation which are arranged predominantly parallel to each other lie perpendicular to the slit of the condenser diaphragm, in which case they would be invisible (Figure 8). It is not until after the preparation has been rotated by 90° that the previously invisible directed elements again appear dark on a bright ground. The azimuth effect also occurs clearly in the case of unordered structures although in less striking fashion. The horizontal or the vertical components are emphasized according to the direction in which the slit-shape diaphragm lies. This phenomenon has already been observed in dark-field microscopy and was pointed out by Siedentopf [2]. Quite conceivably, it could also be put to use for the phase-contrast method in certain cases.

The condenser diaphragm as a substitute source of light is imaged, through condenser and objective, in the exit pupil of the objective. The phase platelet, the thickness of which has been discussed in the theoretical part of this paper, is at this location, and is identical in size and form with the image of the diaphragm. The exit pupil is often located on the inside of the objective. The phase-retarding layer will be arranged on an available lens surface in order to avoid the necessity of a special plate. The phase platelet can now be located either in the cement (*Kitt*) between two lenses or on an air surface and may represent a positive or a negative of the condenser diaphragm. According to circumstances, we obtain a positive or a negative microscopic image as a final result (Figure 9). In this connection, as already stated, we follow Zernike in calling the image positive when elements which are more highly refractive or thicker appear darker than their surroundings. In general, one will prefer the positive phase contrast because of its similarity with the usual bright-field images. Figure 10 may serve to explain this point. Here the same preparation was employed as in the case of Figure 1. In positive phase contrast, the thicker spaces of the grating appear darker, although they have the same penetrability as the deepened slits.

Where bright-field illumination is employed, high-contrast reproduction of objects poor in contrast is further attainable by so adjusting the image that it

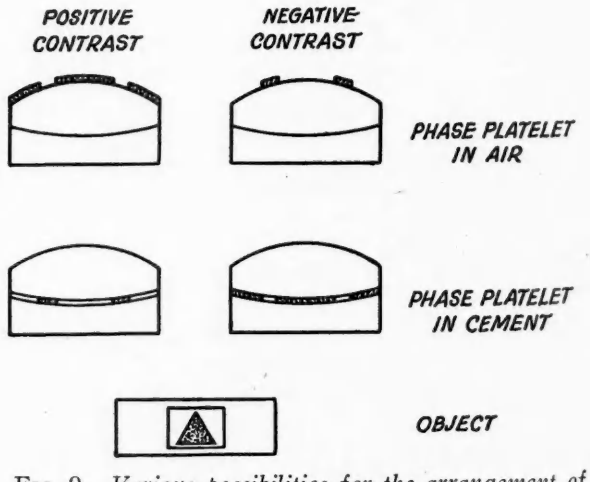


FIG. 9. *Various possibilities for the arrangement of the phase platelet in the objective and their relations to the kind of microscopic image produced.*

is not in true focus. Ordinarily, in the case of such objects for which the preparation of Figure 11 represents a model, the microscope is adjusted to the greatest possible contrast (c). As stated, however, high contrast is obtained at the expense of a loss in sharpness. The desire to see at least something in the image is, even though unconscious, greater than the desire for absolute sharpness. The adjustment to the maximum sharpness (b) produces an image which makes a thoroughly inaccurate impression. Nevertheless it is perfectly sharp, as demonstrated by the photograph, in the case of phase contrast (a). The latter photograph was, in fact, made first, and there is no doubt that it is the sharpest; the bright-field photograph (b) was then made without change of adjustment, and with the aid of the double diaphragm (Figure 16). In the case of c, the tube was depressed by 0.01 mm., while for d, it was elevated by the same amount. It can be seen that the sharp bright-field photograph (b) gives a false picture of the object in that, owing to the diffraction border around the particles, it is not possible to make an exact statement regarding its diameter. Similar observations hold in still greater measure, owing to the occurrence of the Becke lines, for the poorly focused photographs, which simulate positive (c) or negative (d) phase contrast, according to the adjustment.

Hence the phase-contrast method is better adapted than the usual bright-field method to furnish true images of preparations poor in contrast; it also enables adjustment to absolute sharpness so that exact measurements can be made.

The field of application of the phase-contrast method seems to be quite large—at any rate, larger than could be foreseen at the beginning of the development. Of course, nothing impossible should be demanded. Not all objects are equally well suited for this type of study. The principal field is the unstained, sufficiently thin, biological preparation, but

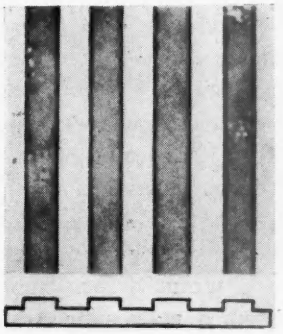


FIG. 10. *Phase grating in the case of positive phase contrast. Underneath, a cross section. The same object as in Figure 1.*

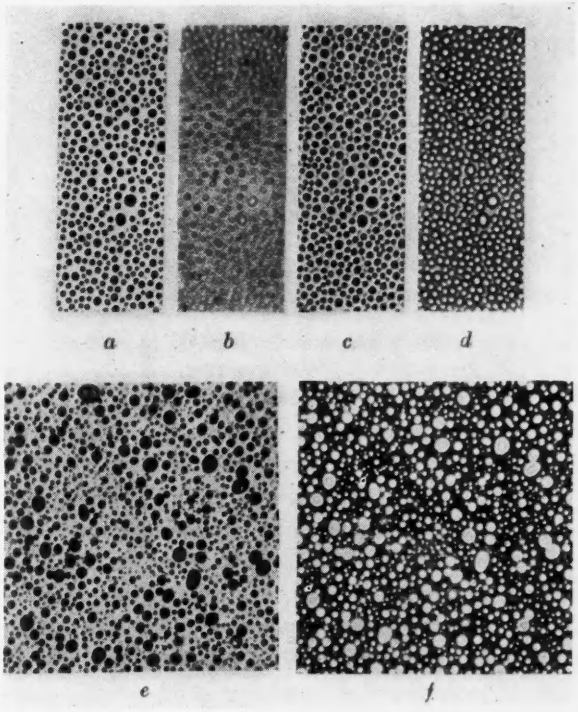


FIG. 11. *Small drop of Canada balsam on glass.*

a–d: *Influence of the adjustment upon the contrast.* a—*Positive phase contrast;* b—*bright field, greatest sharpness;* c—*bright field, tube 0.01 mm. lower than for* a *and* b; d—*bright field, tube 0.01 mm. higher than for* a *and b.*

e–f: *Influence of the diaphragm upon the contrast.* e—*positive;* f—*negative phase contrast obtained by way of positive and negative diaphragm. Approximately 50:1.*

also a few special cases from the technological field can yield suitable objects. A report will be made later regarding the possibility of using the phase-contrast method for the study of unetched metal surfaces. The investigations in this field are still in progress. With the aid of a few figures, we wish to give a general view regarding the various fields of application without laying claim to completeness.

Figure 12 shows a pathological urine sediment. The photograph in the bright field proves unsatisfactory in spite of the reduced condenser diaphragm opening. Only in the center of the field of view are a few large cylinders represented. Fainter cylinders in the center and on the edge are invisible and do not become clear until positive contrast is employed.

The differences in the various methods of imaging can be made clear by taking as an example fresh, diluted blood in the Thoma-Zeiss counting chamber

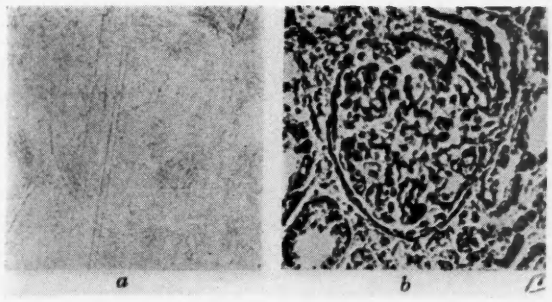


FIG. 14. *Fresh thin section of a kidney in water.* a—*Bright field;* b—*positive phase contrast. 40:1.*

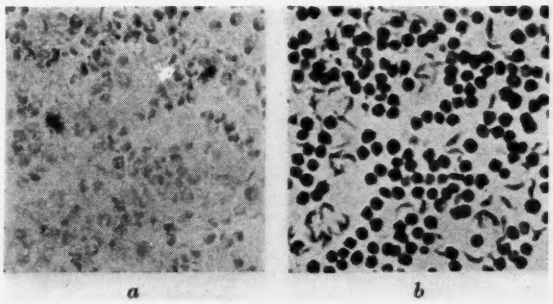


FIG. 15. *Living trypanosomes in the blood of a mouse.* a—*Bright field;* b—*positive phase contrast. 200:1.*

which is separated from the transparent ring by a dark zone. The external edge is also dark. If this filter is used as a condenser diaphragm and if we apply under it a homogeneous herapathite polarizer (c) free to rotate over the entire surface, then the ring and the circle will consecutively appear in the dark position, depending upon the position of the polarizer, as is evident at once from Figure 16. The circle operates like an ordinary iris diaphragm and makes the bright-field photography possible (position I). Slow rotation of the polarizer brings about steady transition to phase contrast, which is complete when the circle is quite darkened and the ring is bright (position II). The time of exposure for both photographs is the same, owing both to the equal area of circle and ring and to the absorption of the phase platelet. It is conceivable that the principle here described could be put to use for other purposes. For example, a double diaphragm is very practical for the adjustment of dark field or light field, as may be desired.

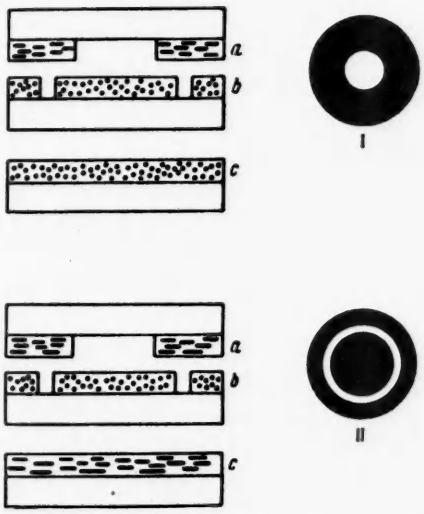


FIG. 16. *Double diaphragm for the transition from bright field to phase contrast.* I—*Position for bright field;* II—*position for phase contrast. The hachures (dashes and dots) indicate the direction of oscillation of the monocrystal layers of herapathite.*

The phase-contrast method is suitable also for higher magnifications with the employment of immersion systems, when suitable objects are available. The limit is set mainly by the thickness of the preparation. The suitability of microtome cuts is dependent on their being especially thin. The embedding medium also naturally plays a great role. Thus the image turns out to be of different quality, depending on whether the object had stood in water or in Canada balsam.

In Figure 17 is represented an epithelial cell of the buccal mucous membrane. The bright-field image shows little more than the contours of the cell. Of the cell nucleus, almost nothing is to be seen. Of course, it might have been pictured more clearly if the photograph had not been made in such exact focus. This point has already been made in connection with Figure 11. The phase-contrast picture, on the contrary, is scarcely distinguishable from the view of a well-stained preparation. The nucleus appears with utmost clarity, as do also the clumps and corpuscles distributed rather irregularly in the plasma. On the border of the cell may be faintly recognized the fine toothing of the cement, which, on being set on the surface of the cell, covers over the entire cell body as a fine lattice work.

It is worthy of note that the cell nucleus of white blood corpuscles is reproduced bright, not dark, as is shown by Figure 18. Here also, for a magnification of 1000, the contrast effect is still quite good. The red corpuscles appear as dark discs with the central portions more transparent than the edges. The incipient erythrocyte formation with protruding spicules (*Stechapfelbildung*) is also readily recognizable.

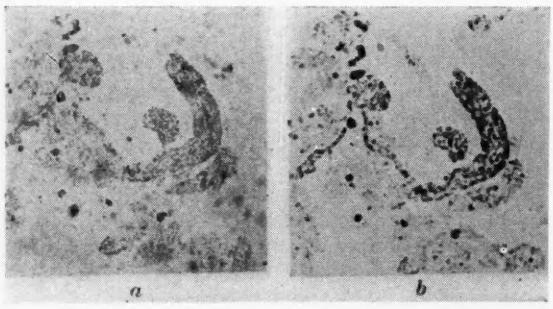


FIG. 12. *Pathological urine sediment.* a—*bright field;* b—*positive phase contrast. 80:1.*

(Figure 13). In the case of bright-field photographing (a), the red blood corpuscles appear as darkly contoured circles; the lines of the network (*Netzteilung*) of the chamber are likewise dark, partly invisible. On the whole, the picture is faint and lacking in contrast when compared with the adjoining picture, in which positive phase contrast was employed. Here the blood corpuscles are reproduced essentially darker and stand out with unequal clarity from the background. The lines of the network, which of course are depressions in the glass, appear brighter than the surroundings, as is to be expected from theory. The negative phase-contrast picture (c) is an exact negative of the previous picture (b).

In the case of the dark field, at first one is reminded of the picture with negative phase contrast. There are, however, essential differences. The background here is pure black, as it cannot be in the case of negative phase contrast. With the dark field the erythrocytes are represented mainly by their bright contours, whereas in phase contrast they are bright discs. The net division for the dark field is bright; for negative phase contrast, dark. The phase-contrast picture, negative or positive, reproduces the natural relations much more correctly than the bright-field or the dark-field one. The red blood corpuscles are of course not rings, but corporeal discs, and they are also represented as such by the phase-contrast method.

The histological unstained sectional preparation of a kidney shows the well-known undifferentiated appearance of such pictures in the case of bright-field illumination (Figure 14). On the contrary, the picture of the same preparation in positive phase contrast looks very rich in contrast and at first glance is indistinguishable from that of a stained preparation. A somewhat more exact analysis of such pictures shows that in them are represented prodomi-

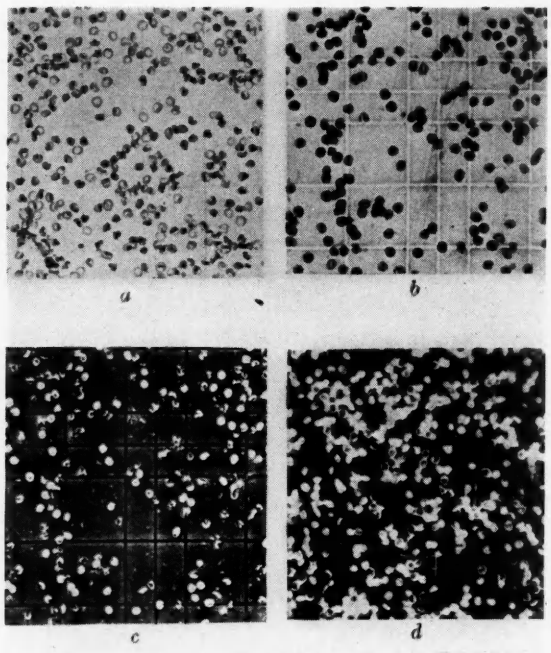


FIG. 13. *Fresh blood in the Thoma-Zeiss counting chamber.* a—*Bright field;* b—*positive phase contrast;* c—*negative phase contrast;* d—*dark field. 160:1.*

nantly elastic fibers, cell walls, and, above all, cell nuclei. The section was obtained with the freezing microtome and immediately placed in water between cover glass and slide. The time between the taking of the material and the microscopic observation amounted therefore only to a few minutes. This opens up the possibility of making histological diagnoses in a much shorter time than has hitherto been the case.

The attempt to represent living trypanosomes in the blood was successful beyond expectations. We were able to produce a short strip of film, a cutting of which is shown in Figure 15.

The film was photographed in a special manner. In order to show clearly the difference between the usual bright-field illuminaiton and phase contrast, we endeavored to obtain a continuous transition from the one to the other mode of observation. This was accomplished as follows. A circle is cut out from the center of a monocrystal layer of herapathite (Figure 16, a). From a second layer (b) a ring is cut out concentrically around the circle. The areas of these two figures are equal. The polarization filters are so cemented together that their directions of oscillation are mutually perpendicular. The new filter thus formed reveals the transparent circle in the center

lower focal plane of which ring diaphragms of various diameter, suitable for the different objects, can be applied. The diaphragms must be centerable and adjusted in height so that their pictures can be imaged sharply in the objective so as to cover the phase platelet. For the correct adjustment of the diaphragm image, there is also needed an auxiliary microscope, which can be inserted in the microscope tube in the place of the ocular, enabling the observation of the diaphragm image and phase platelet in magnified form. After adjustment has been effected, the auxiliary microscope is exchanged for the ocular.*

Summary

By way of logical development of the Abbé theory of microscopical representation, Zernike was able to show that through arbitrarily influencing the phase of a part of the light an increase of the contrast in the microscopical image could be attained. The theoretical basis of this phase-contrast method has been discussed in detail, and reference has been made to the reasons for the employment of circular, ring-shaped phase platelets. By the way of illustration, the fundamental differences between bright field and dark field and between positive and negative phase contrast are discussed. It is shown that in many cases the new method is fundamentally superior to the dark field and the light field; it furnishes images more like the object than do the latter methods. Unstained, even living microscopic preparations can be observed and photographed in a particularly successful manner and with a clarity not hitherto regarded as possible.

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* The accessories required for carrying out the process will be put on the market by the Carl Zeiss firm in the foreseeable future.

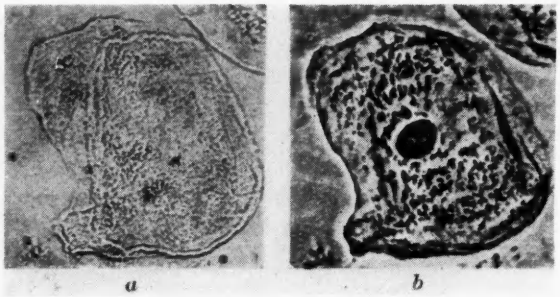

FIG. 17. *Epithelial cell of the buccal mucous membrane.* a—*Bright field;* b—*positive phase contrast. 500:1.*

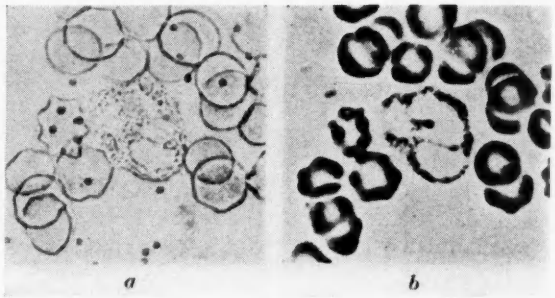

FIG. 18. *Fresh human blood. In the center a leucocyte.* a—*Bright field;* b—*positive phase contrast. 1000:1.*

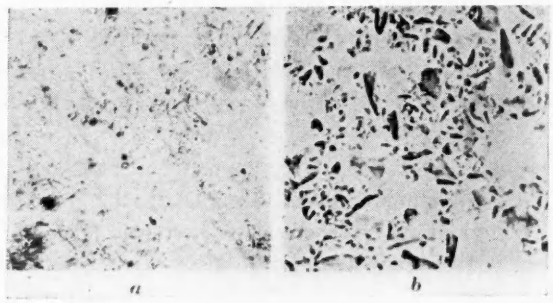

FIG. 19. *Glass powder in liquid.* a—*Bright field;* b—*negative phase contrast. 80:1.*

It is a striking fact that the diameter of the erythrocytes in the bright-field image is 10 to 15 percent greater than in the contrast picture. This leads one to suspect that the previous measurements on fresh blood are erroneous and stand in need of revision.

The embedding method for determining the refractive index of solid substances rests, as we know, on having the substance finely divided and brought into a liquid medium, the refractive index of which is constantly changed through change of concentration or temperature until, under microscopic observation, the particles of substance and their contours can no longer be seen, and hence until the light refraction of the two substances is the same. The refractive index of the liquid is then measured with the refractometer, due consideration being given to the temperature. The value thus obtained is the value sought for the solid substance. The Becke method permits in practice an accuracy of measurement of 0.001 in case the microscopic observation was undertaken with monochromatic light and the highest possible magnification. Kofler [8] has recently announced an accuracy of 0.0002. Even for the determination of melting point as described by him, the phase-contrast method might bring with it an increase of accuracy. According to the observations thus far made, the hope for an increase of accuracy through application of the phase-contrast method now appears justified. In Figure 19, a case has been selected at random. In the bright field, the requirement of the Becke method—namely, the lowest possible visibility of the particles—seems to be fulfilled. The phase-contrast method reveals, however, that no absolute agreement of the refractive index yet exists. The fact that a positive image arises in spite of the employment of negative phase contrast is an indication that here the enclosing medium has a higher refractive index than the particles.

The described method of phase shifting is furthermore of great importance for the method of Boersch [9, 10] for rendering visible sub-microscopic crystal lattices.

In this method, the diffraction figure of a crystal lattice is produced artificially in the upper focal plane of the objective through blinding out (*Ausblenden*). In this way the image of the crystal lattice belonging to this diffraction figure is simulated in the projection plane of the microscope, although the microscope contains no object resembling a lattice. The figure thus produced represents the roentgen diffraction diagram of the crystal.

In order to show crystal lattices whose diffraction reflexes (diffraction maxima) are mutually shifted in phase, the reflexes produced in the microscope through blinding out must also be mutually shifted in phase. This phase shifting can be undertaken in accordance with the method described.

For the practical carrying out of the phase-contrast method the principal need is for microscope objectives which are equipped with the phase platelet. Such objectives can also be employed without disadvantage for ordinary bright-field and dark-field microscopy. Furthermore, a condenser is required in the

Some Comparative Data on the Cross-Sectional Swelling of Textile Fibers

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ONE of the important properties of a textile fiber is its behavior towards water. If the fiber absorbs water it swells and this swelling affects not only the fiber dimensions and physical properties but also the general reactivity of the fiber.

Most fibers swell in cross section more than in the longitudinal direction, as shown in the following tabulation:

Fiber	Longitudinal swelling [1] (%)	Cross-sectional swelling (%)
Cotton	1.1	20–26
Wool	1.2	22–26
Silk	1.3–1.7	19–20
Rayon	0.7–7.0	44–86

A search of the literature reveals a dearth of comparative data on the cross-sectional swelling of the common textile fibers. It is the purpose of this paper to present some data which we have obtained by measuring the cross-sectional areas from photomicrographs. Since the work is being continued, no attempt will be made to interpret the data in relation to fiber structure or properties. The data and technique are offered at this time with the hope that they may prove useful to other textile technologists.

Method

The swelling data presented in this paper were obtained by measuring the increase in area of the fiber cross section when swollen in water. The percent swelling is calculated as follows:

$$\frac{\text{Swollen area} - \text{Dry area}}{\text{Dry area}} \times 100 = \%\ \text{Swelling}$$

The area measurements are made from photographs of the same fiber in both the wet and the dry condition.

Procedure: Cross sections 10 μ thick are prepared by the paraffin method. For the dry-area measurement, the sections are conditioned in the laboratory, mounted in *n*-heptane or other nonswelling media, and a selected field is photographed (Figure 1). The *n*-heptane is then allowed to evaporate, is replaced by water, and the same field is rephotographed wet (Figure 2). All photomicrographs from which the data in this paper are calculated were made with the oil immersion objective. Proper precautions were taken in the illumination of the microscope for maximum optical performance, and the resolution was further enhanced by the use of a shorter wave-length portion of visible light (Wratten C and H filters, average wave length 460 mμ).

The final prints were enlarged photographically to 5,000 diameters. This relatively great magnification assists materially in the measurement of fiber areas. The areas were determined either with planimeter or by cutting out and weighing the photographic images of the filaments on an analytical balance. In either case, the areas of the same fiber or filament in the swollen and dry states were compared, showing an accuracy of measurement to about 0.2%, depending upon the size of the cross section. However, these differences are largely compensating.

Most of the swelling measurements previously reported in the literature have been determined from an average longitudinal width, with a circular or square cross-sectional shape assumed. In the present method, the comparison of areas of the same identical cross section eliminates the variable of fiber size and takes into account irregularities of fiber shape.

Results

In Tables I–VI the results of the measurements of these specially prepared cross sections are given. The variation in the swelling of individual filaments

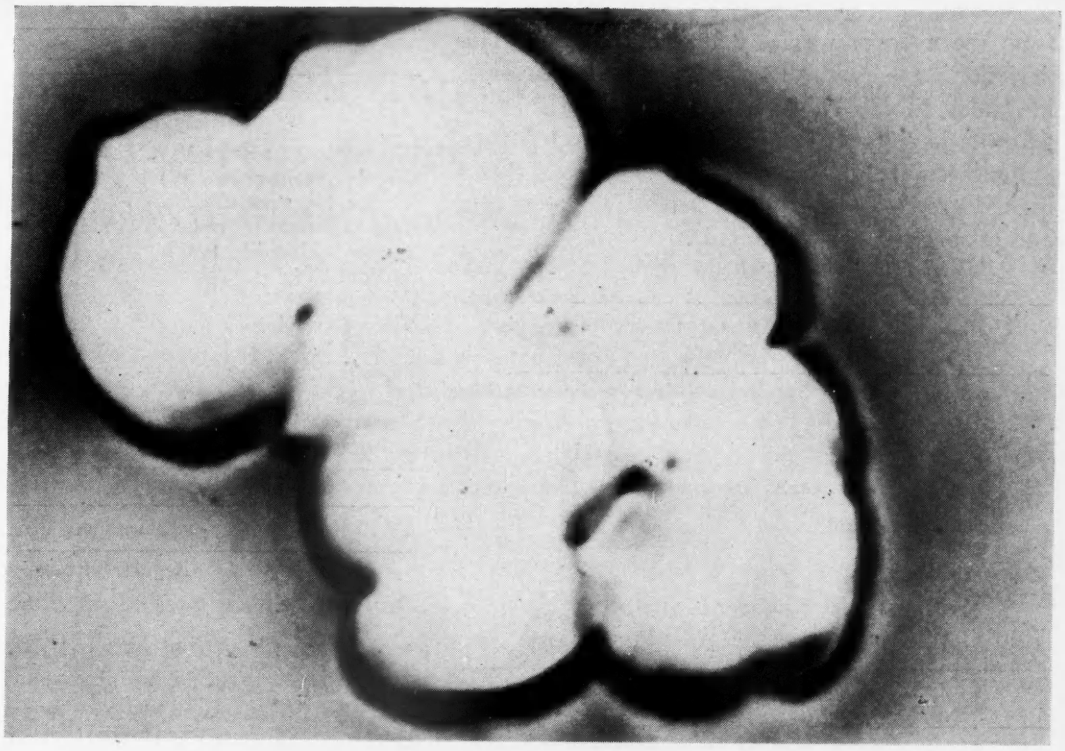

FIG. 1.
"Strong type" rayon filament mounted dry in n-*heptane. Magnification 5000 ×.*

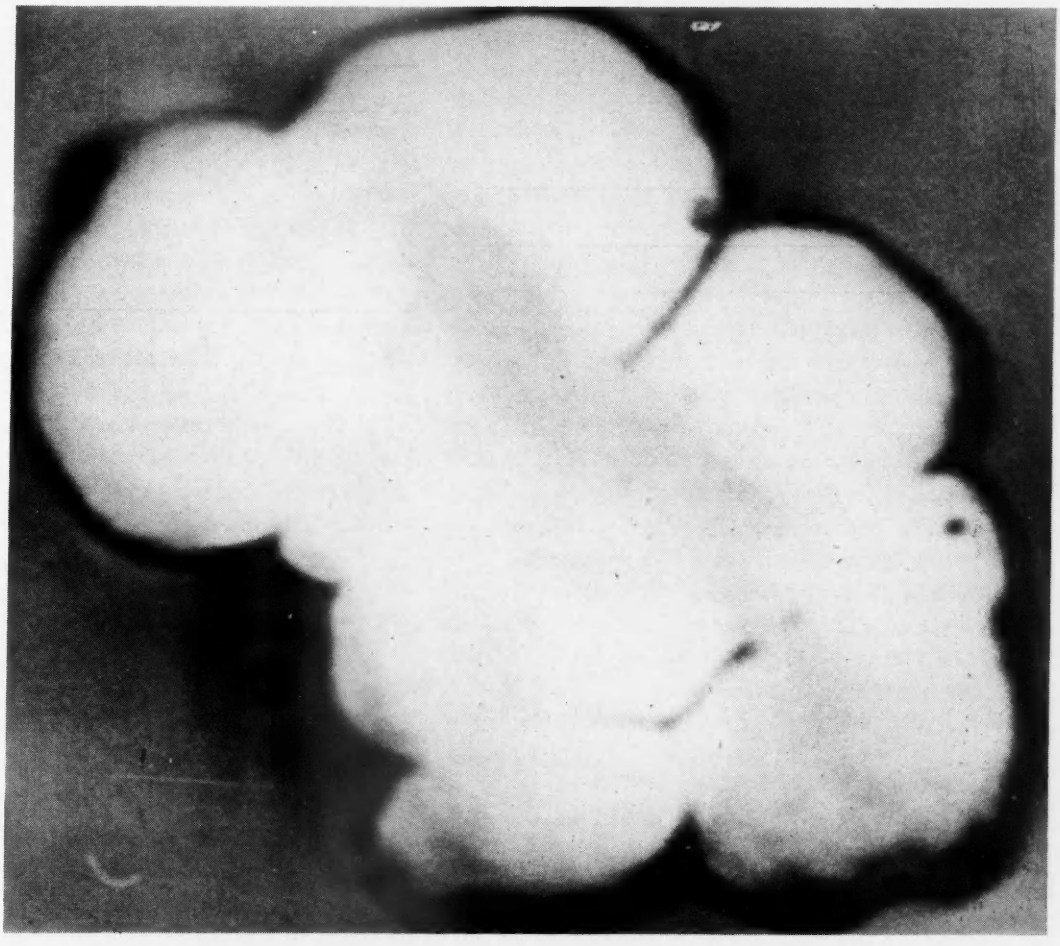

FIG. 2.
Same filament as Figure 1. Swollen in water. Magnification 5000 × (swelling 52.3 percent).

from the average was ± 2.5 percent mean. This deviation varied with the type of fiber; for example, in Aralac (Table I) the mean deviation was ± 0.2 percent; in cotton (Table III) it was much higher.

For accurate measurements, it is obvious that a high degree of perfection in sectioning, mounting, and photographing is required.

TABLE I. FIBERS OF ANIMAL ORIGIN (INCLUDING CASEIN FIBERS)

Fiber	Cross-sectional swelling (%)
Silk	19
Domestic wool	26
Australian wool	25
South American wool	26
Dress fabric wool	26
Blue serge wool	22
Wool 48–S	24
Aralac 70–S	18.8
Aralac 50–S	18.9

TABLE II. FIBERS FROM SYNTHETIC POLYMERS

Fiber	Cross-sectional swelling (%)
Nylon	3.2
Nylon stocking	1.6
Vinyon CN	4.3
Vinyon CF	0.2
du Pont Fiber A	5.1

TABLE III. THE NATURAL CELLULOSE FIBERS

Fiber	Cross-sectional swelling (%)
Cotton	21
Mercerized cotton	24
Flax tow	47
Bleached ramie	37

TABLE IV. THE RAYONS

Fiber	Cross-sectional swelling (%)
Viscose (high tenacity)	50.0
Viscose (regular tenacity)	65.0
Viscose (pigmented)	66.6
Cuprammonium (Bemberg)	56.2
Viscose (Lilienfeld type)	44.8
du Pont Fiber G	37.1
Fortisan	22.0
Viscose (staple)	63.4
Rayolanda	52.0
Slovolna	59.6
Cisalpha	64.6
Acetate (bright)	7.9
Acetate (pigmented)	7.9

TABLE V. EFFECT OF DENIER PER FILAMENT ON SWELLING

Regular viscose	
Den. per fil.	Cross-sectional swelling (%)
10.7	50.9
5.0	54.1
3.0	56.7
1.67	62.4
1.25	66.0
1.00	63.6

TABLE VI. RESIN TREATED VISCOSE RAYON

Treatment	Cross-sectional swelling (%)
Original	76.9
Rhonite 609	32.8
Aerotex	38.6
Resloom	43.1
Glyoxal	56.2

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Predicting Equilibrium Moisture Relations
With Particular Reference to Textile Fibers*

John C. Whitwell† and Richard K. Toner‡

Contribution of the Textile Research Institute and the Department of Chemical Engineering, Princeton University

In a previous paper [5] of this series, it was indicated that methods of forecasting equilibrium data would be subsequently presented. It is therefore the purpose of this paper to discuss the methods used in extrapolating experimental equilibrium moisture regain data and to outline the procedures by which an entire family of equilibrium curves may be predicted with a minimum of experimental data.

Determining Intercept Values

It has been shown [8] that it is possible to replot moisture equilibrium data which are usually presented as in Figure 1, in the Othmer form (Figure 2) [4]. This is done by drawing isosteres (constant moisture regain lines—that is, horizontal lines on Figure 1) and reading the relative humidities at the intersection of the isosteres with the isotherms. The vapor pressure of water ($P°$) at each of the temperatures may be obtained from a standard reference, such as Lange [1]. Multiplication of each of these values of $P°$ by the corresponding relative humidities (from Figure 1) gives the equilibrium partial pressures (P) of water above the samples. Figure 2 results when the logarithm of $P°$ is plotted against the logarithm of P. The data for these lines, when treated by statistical methods [2], customarily show probability of intersection in a common point.

The intersection of the isosteres with a given $P°$ results in a series of points referred to as "intercept" values.§ This procedure is equivalent to determining the values of P at a specific temperature, since $P°$ is determined by the temperature.

Plotting Intercept Values

The form of intercept plot that will be most convenient for extrapolation to regions not thoroughly covered either by experiment or statistical analysis of the data available would be a straight line. Figure 3 shows such an extrapolation plot to low regain regions. It includes the data for purified cotton by Wiegerink [10] in the normal region and by Neale and Stringfellow [3] in the very low regain regions. It will be observed that although the line curves appreciably at higher and lower moistures, it has a nearly linear intermediate section where experimental data are frequently unavailable. Since Neale and Stringfellow in purification treated their cotton by a bleach followed by a wash with dilute ammonia, their method is analogous though not identical with that of Wiegerink, in which purified cotton was prepared from raw cotton yarn by successive extractions with alcohol, ether, and 1-percent NaOH solution, followed by rinsing in 5-percent acetic acid solution and in water. Unfortunately, Neale and Stringfellow are the only experimenters who have made a thorough investigation of moisture regain under these extremely low moisture conditions. Until additional data are available, it is necessary to assume that the behavior of other materials will be similar to that of cotton; certainly the evidence from the normal regions indicates that this assumption is justifiable.

Interpretation of Curve Families

Inspection of the intercept plots for different wools and silks (Figures 4 and 5) reveals certain conditions of similarity. In the linear section, the lines

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* This paper is the fourth of a series. In the text references are made to previous papers which appeared in this journal.

† Associate Director of Research, Textile Research Institute, and Professor of Chemical Engineering, Princeton University.

‡ Member of the Textile Research Institute Staff and Assistant Professor of Chemical Engineering, Princeton University.

§ The true intercept value occurs only when $P° = 1$ (log $P° = 0$). This designation of "intercept" has been used consistently in the present work.

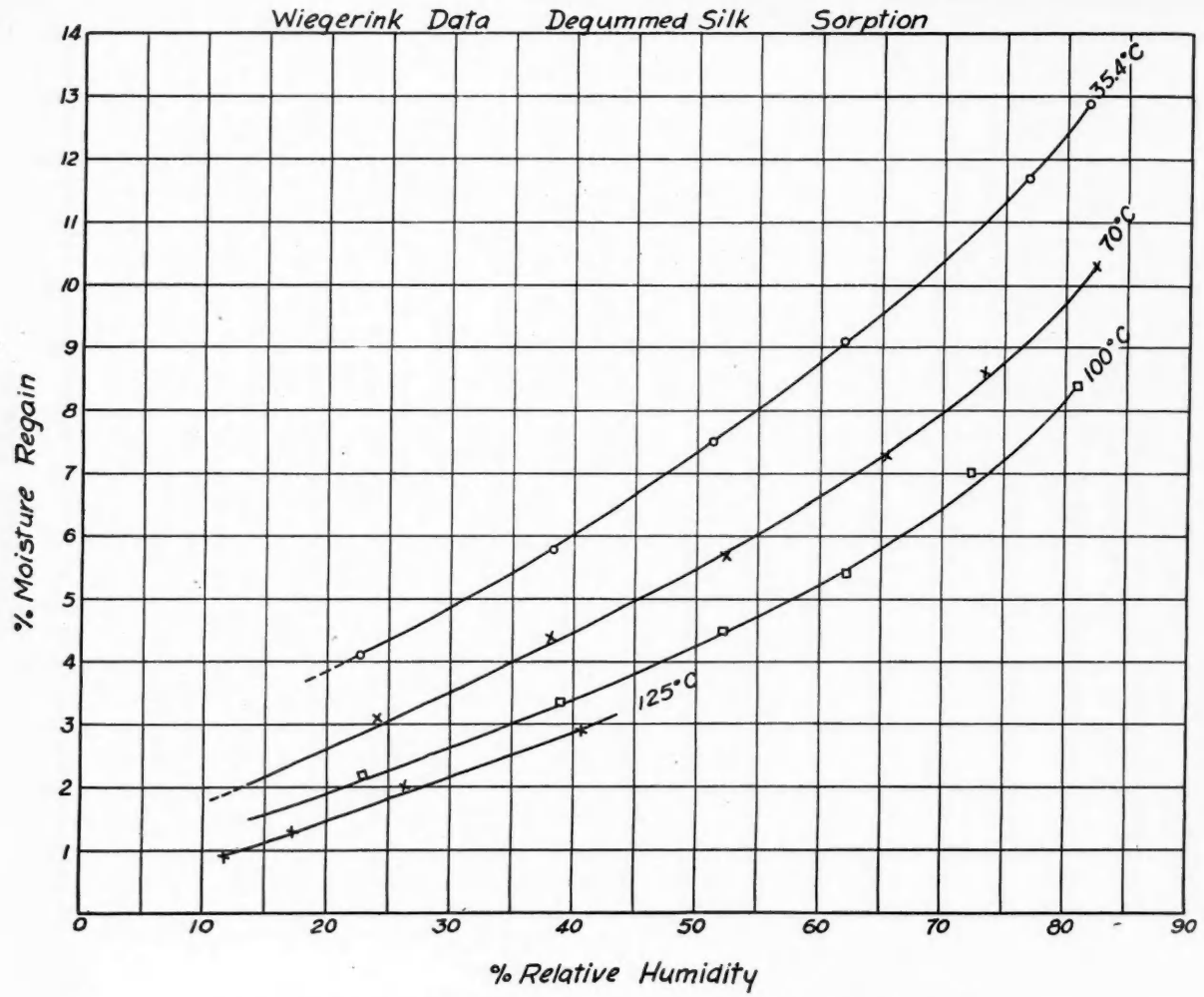

FIG. 1. *Degummed silk in sorption. Original data of Wiegerink.*

actually become parallel for analogous materials. This observation will later be seen to be of considerable value in determining the proper form of the curve from limited data. Care must be taken not to extend this principle to materials which are not truly analogous. For example, mercerized cotton cannot be expected to behave as raw cotton nor should viscose rayon and cuprammonium rayon be expected to act in an exactly equivalent fashion. Despite these variations between materials, similarity of intercept curve form always exists within any one material for both sorption and desorption lines. In Figures 6 and 7 this point is particularly evident. Thus, although the lines for mercerized and purified cottons are different, the sorption-desorption lines for each material are seen to be very similar. The same is true for the cuprammonium and viscose rayon lines.

Use of Intercept-Regain Curves

Extrapolation to Lower Moisture Regains

Once the intercept curve has been established, it can, with reasonable accuracy, be extended to moisture regains lower than those covered by the experiments. The intercepts so determined can then be plotted as log P° vs. log P and these points in turn can be connected with the common point of intersection. Thus the form shown in Figure 2 is obtained. The lines so drawn are constant moisture lines; from them may be read the vapor pressure over the fiber at any temperature. By reversing the calculations used to obtain Figure 2 from Figure 1, it is possible to convert the extrapolated data to the usual form of moisture regain vs. relative humidity.

If one plots the slope of the constant moisture lines from the log P° vs. log P curve as a function of

moisture regain, it is found experimentally that for cotton a curve of the form of Figure 8 is obtained. As has been pointed out in the previous paper [8], constant moisture regain lines at very low relative humidities are parallel; therefore, their slopes are equal. The horizontal part of Figure 8, from the data of Neale and Stringfellow [3], illustrates this condition. On the other hand, as the moisture regain increases, the behavior approaches that of pure water (that is, the curve becomes asymptotic to a value of 1.0). Between these two limits there is an indeterminate range which needs further data for clarification. In this transition region, the Othmer lines change from a parallel grouping to one in which there is a gradation in slope resulting in an intersection in a common point. This transition occurs in a low moisture regain region and therefore may not be of too much practical importance.

Extrapolation to Higher Moisture Regains

In general there are more experimental data in the high regain region and therefore the need for extrapolation is less. The type of extrapolation curve previously described is not of much use here. However, if the logarithm of the intercept is plotted against the actual value of the moisture regain rather than against the logarithm of this value, a suitable form is obtained. Figure 9 is an example of this type of extrapolation curve. Inspection of the curves shows an approach to the intercept value of pure water, the curves becoming asymptotic to the value zero. Once established, this curve is used in the same manner as the extrapolation curve for lower regions.

It is somewhat questionable how high the extrapolation may be carried. Physical changes may occur in the fiber which alter its characteristics. For example, it will be noted in the data of Urquhart and Williams [6] that the isotherms for the higher temperatures cross those for lower temperatures when relative humidity is high. This behavior is not corroborated by the Wiegerink [10] data, but it must be remembered that the two sets of data were not taken under similar conditions. Moreover, at higher temperatures and relative humidities the physical measurements might reasonably be expected to be less reliable. If appreciable swelling or other physical modification materially alters the fiber, predictions based on normal behavior would no longer hold true. Decision regarding the ultimate top limit for the method cannot be made with the data at hand.

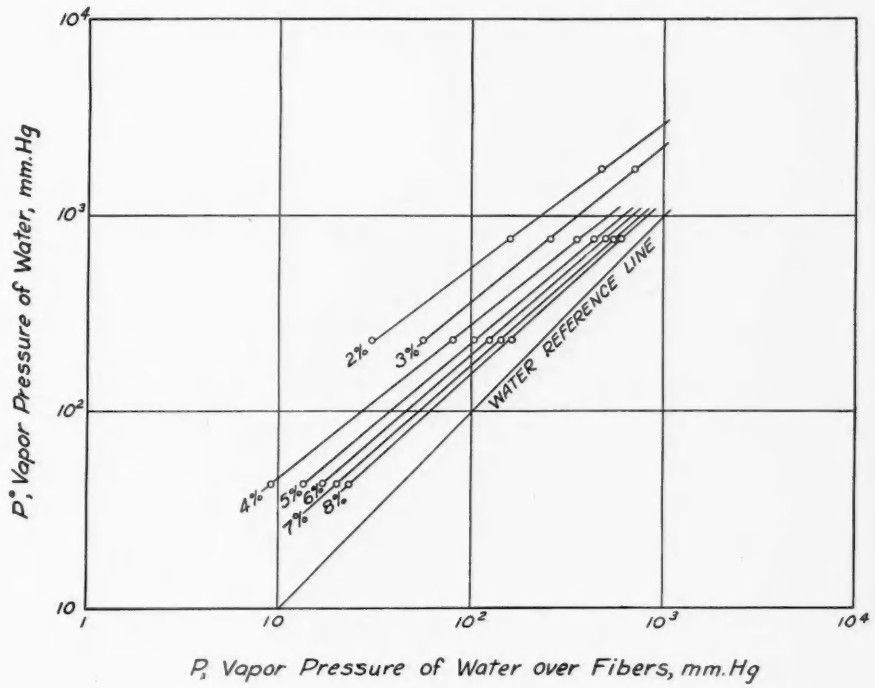

FIG. 2. *Othmer plot derived from Figure 1.*

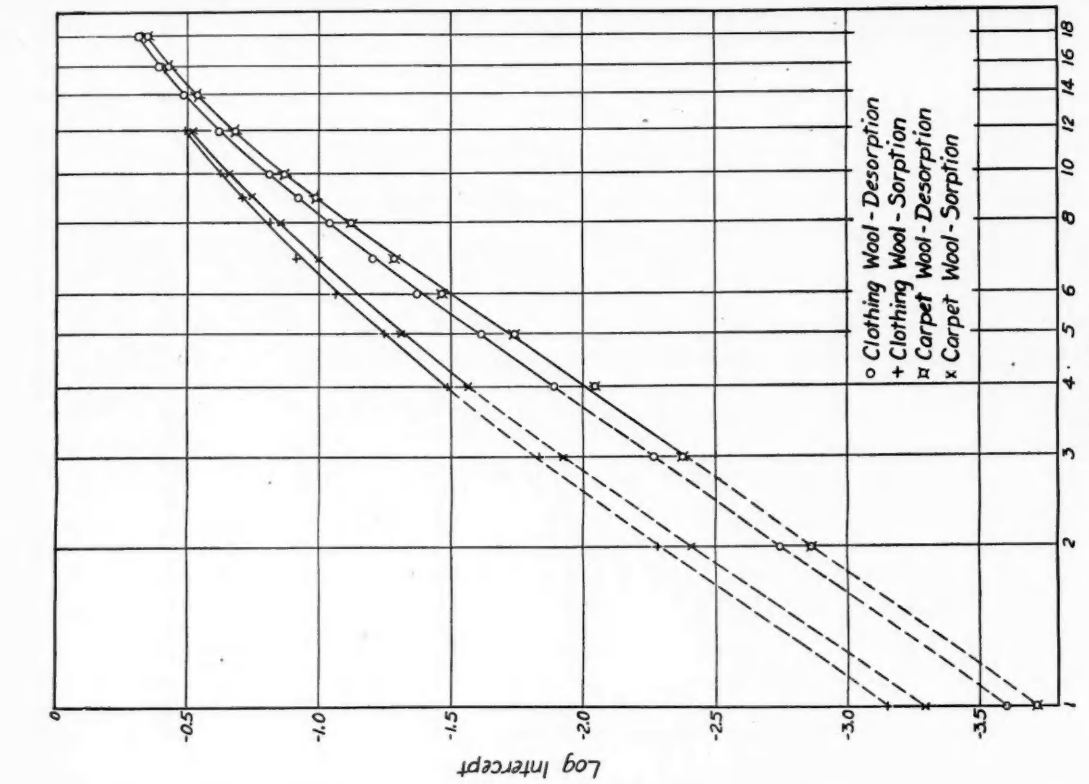

FIG. 4. *Intercept curve, wools, sorption and desorption.*

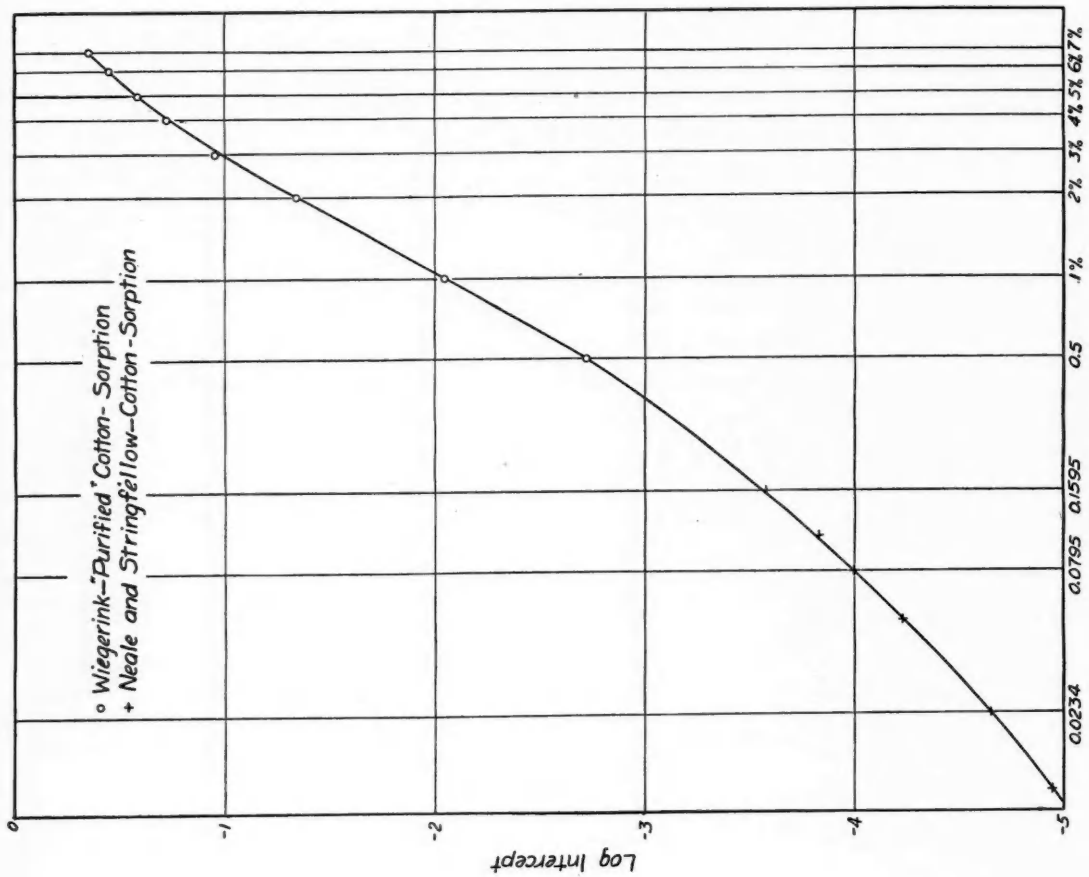

FIG. 3. *Intercept curve, cotton, sorption.*

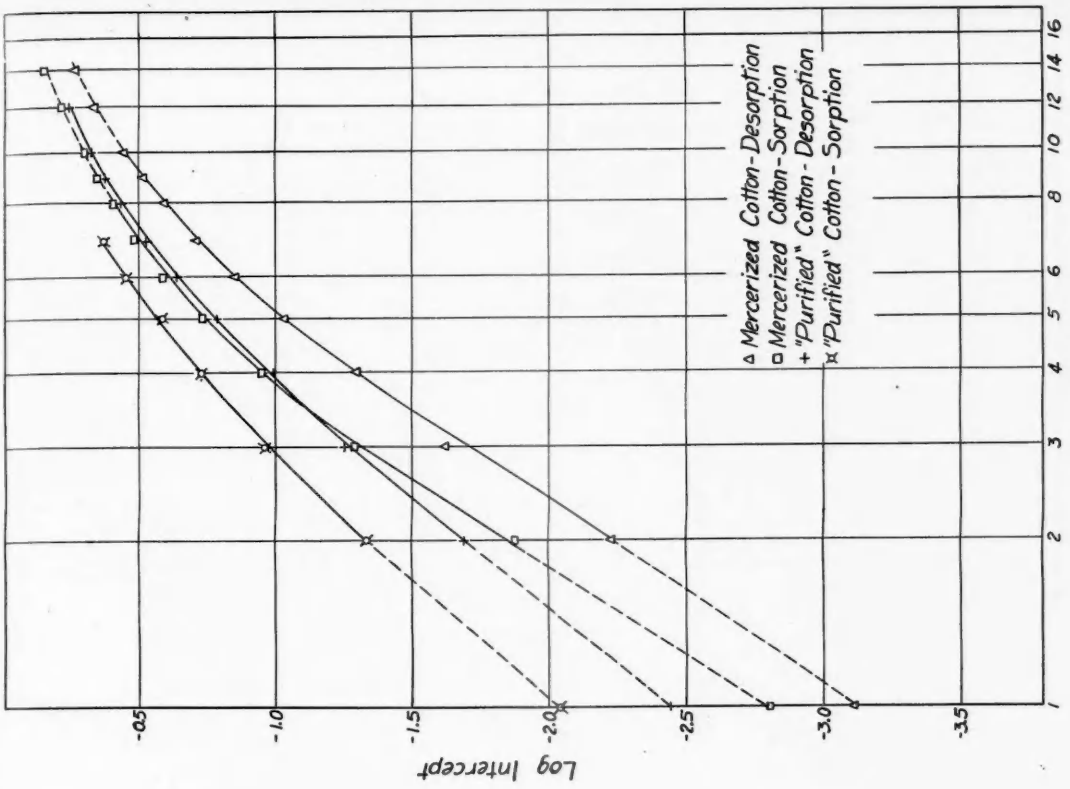

FIG. 6. *Intercept curve, mercerized and purified cottons, sorption and desorption.*

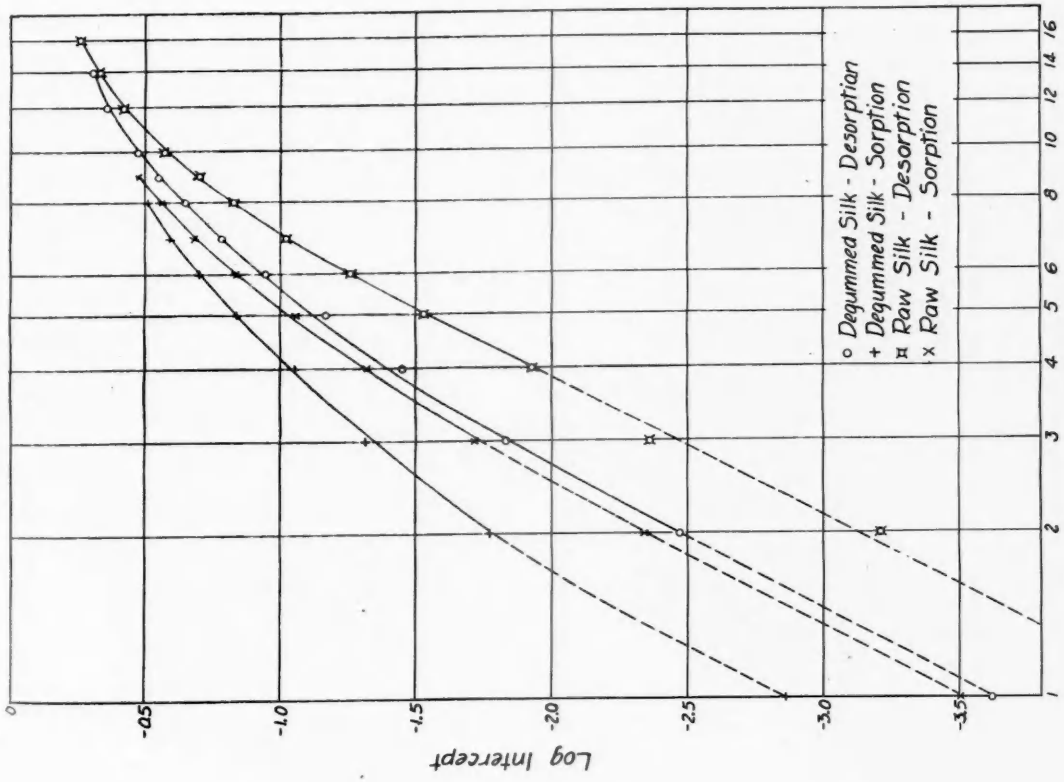

FIG. 5. *Intercept curve, silks, sorption and desorption.*

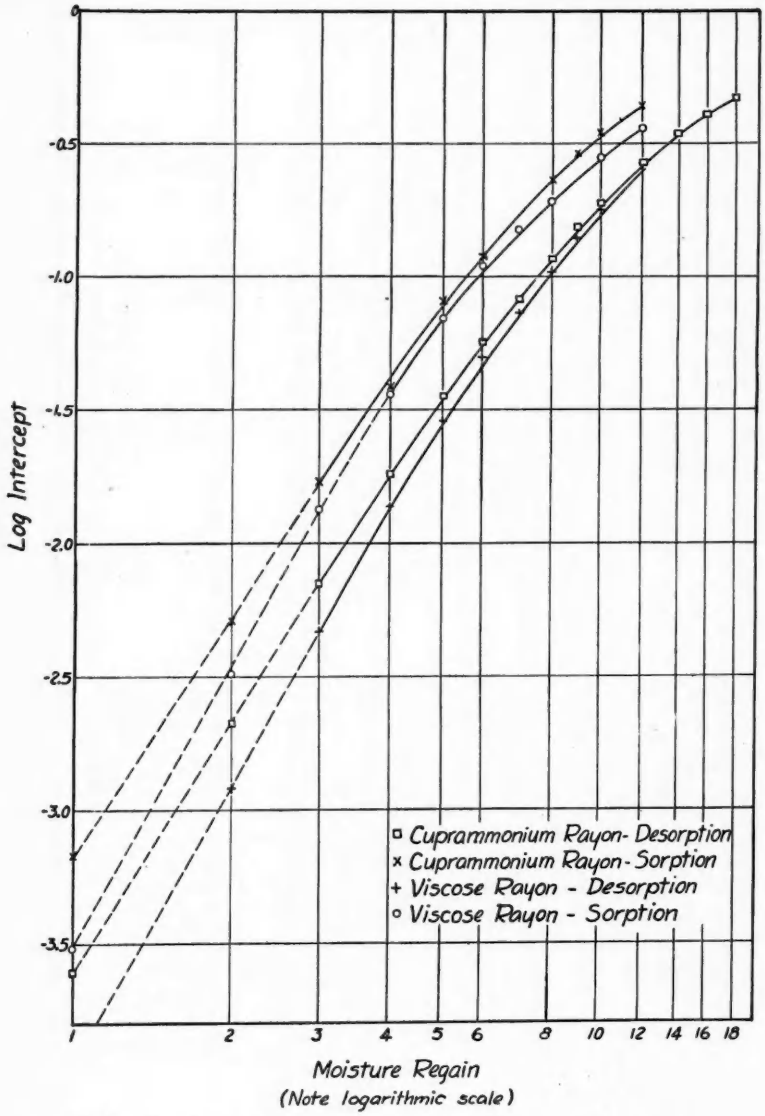

FIG. 7. *Intercept curve, cuprammonium and viscose rayons, sorption and desorption.*

Extrapolation to Other Temperatures

Inasmuch as values of $P°$, the vapor pressure of pure water, are directly dependent upon the temperature, the Othmer plot may be conveniently used to obtain the regain-relative humidity curve for any desired temperature. If one extends the straight isosteres to the desired lower temperatures (lower $P°$ value), the values of P may be read. The relative humidity is obtainable as usual, by dividing P by $P°$. This procedure has been performed by the authors in a comment on other work in the moisture field [7].

The lower limit of temperature to which the method is applicable would be expected to be 0°C, the freezing point of water. While all the water associated with the fibers may not freeze below this temperature, a portion of it would be expected to do so [7], resulting in a change of slope of the isostere [9].

Predicting Moisture Equilibrium Relations

Prototype of Fiber Previously Analyzed

The basic principle used in this method is that of similarity of curves for the same type of material despite differences in actual values due to previous history of the fiber. This similarity, noted earlier, is particularly important in the intercept-regain relations. To predict moisture regain relations, it is necessary first to determine in the laboratory a value for a sample under known relative humidity and temperature conditions. This point may then be recorded on an Othmer plot; the common point of intersection for the material is also plotted, as obtained from Table I. These two points may then be connected since constant moisture regain conditions are represented by a straight line. Extending this line to $P° = 1$, the intercept for the experimental condition is obtained.* The logarithm of its value is then plotted on a figure of the type of Figure 3 and a new intercept curve is drawn through it similar to the established curves for that material, parallel in the low moisture regain region and converging in the higher region.

* All the work up to this point may more suitably be done by calculation than by plotting curves.

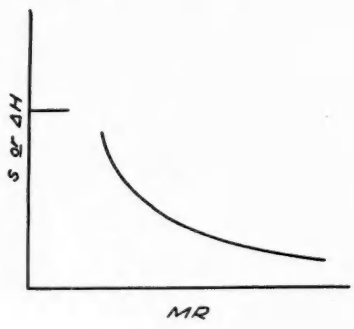

FIG. 8. *Slope (heat effect) of Othmer lines vs. moisture regain.*

With this new intercept curve, it is possible to obtain a series of intercept values at various moisture regains which can then be plotted on the log P vs. log P° graph on which the experimental point has already been located. Each of these points in turn may be connected with a common point of intersection. All information necessary to recalculate a complete set of equilibrium curves is then available.

Since this method is naturally subject to experimental errors and variations in samples, it can be improved by locating the intercept curve for more than one sample.

ILLUSTRATION

As an example of this method, wool samples were stored over sulfuric acid of known concentration until they reached equilibrium. They were then analyzed by the standard ASTM procedure and the results given in Table II were obtained. These values were next plotted on an Othmer plot on which the location of the common point for wool is known. On Figure 10, points A, B, C, D, and E represent the results of the laboratory determinations. Each of these experimental points has been connected with the intersection point (not shown in Figure 11 because of space limitation) to give a straight line representing the average moisture regain of the samples so conditioned. If these lines are now extended to $P^\circ = 1$, intercept values (A′, B′, C′, D′, E′) may be read and plotted, as in Figure 11. It will be noted that the curve (Figure 11) through the experimental data is similar to that obtained for the Wiegerink wool data [10]— parallel in the lower regions and converging in the higher regions. This curve may now be used to pick off a smoothed intercept value corresponding to any moisture

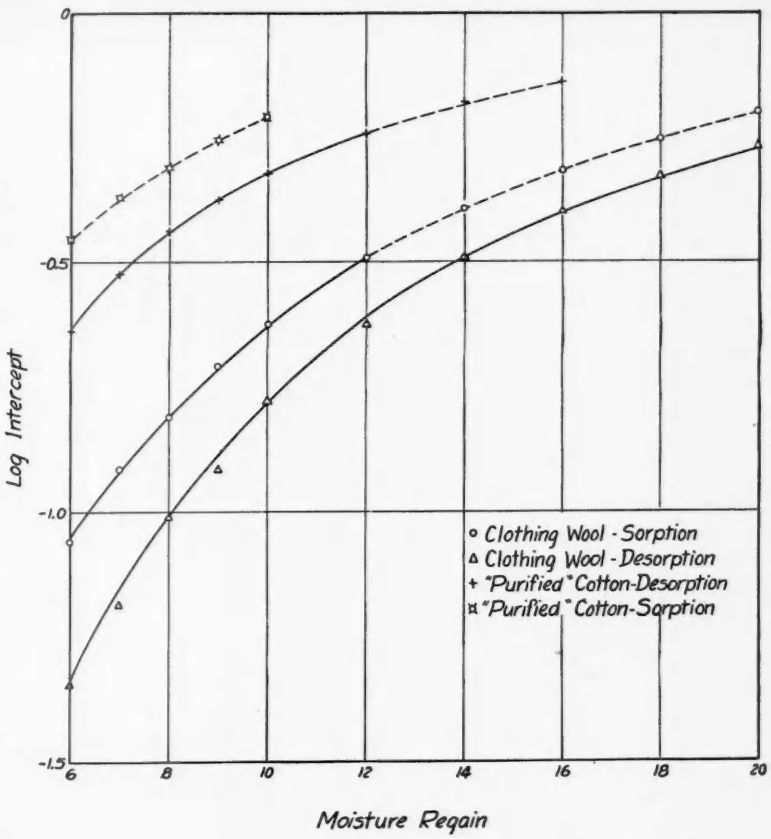

FIG. 9. *Intercept curve, purified cotton and wool—for high regain region only.*

TABLE II. EXPERIMENTAL DATA ON DYED WOOL TOPS, 50°C

Sample	RH (%)	MR (%)
A	3.3	1.80
B	3.7	1.66
C	14.4	3.74
D	27.2	5.66
E	34.7	6.89

TABLE I. COMMON POINT VALUES*

	Desorption		Sorption	
Purified cotton	$P^\circ = 3.16 \times 10^5$	$P = 4.79 \times 10^5$	$P^\circ = 26.91 \times 10^5$	$P = 52.00 \times 10^5$
Raw cotton	$P^\circ = 1.35 \times 10^5$	$P = 1.86 \times 10^5$	$P^\circ = 3.16 \times 10^5$	$P = 5.50 \times 10^5$
Mercerized cotton	$P^\circ = 1.47 \times 10^5$	$P = 2.19 \times 10^5$	$P^\circ = 2.00 \times 10^5$	$P = 3.02 \times 10^5$
Raw silk	$P^\circ = 0.32 \times 10^5$	$P = 0.46 \times 10^5$	$P^\circ = 1.00 \times 10^5$	$P = 1.48 \times 10^5$
Degummed silk	$P^\circ = 0.65 \times 10^5$	$P = 1.07 \times 10^5$	$P^\circ = 2.95 \times 10^5$	$P = 5.89 \times 10^5$
Carpet wool	$P^\circ = 3.16 \times 10^5$	$P = 6.31 \times 10^5$	$P^\circ = 3.55 \times 10^5$	$P = 6.76 \times 10^5$
Clothing wool	$P^\circ = 4.17 \times 10^5$	$P = 7.76 \times 10^5$	$P^\circ = 7.94 \times 10^5$	$P = 16.60 \times 10^5$
Viscose rayon	$P^\circ = 1.58 \times 10^5$	$P = 2.51 \times 10^5$	$P^\circ = 10.00 \times 10^5$	$P = 19.06 \times 10^5$
Cuprammonium rayon	$P^\circ = 3.63 \times 10^5$	$P = 6.92 \times 10^5$	$P^\circ = 1.51 \times 10^5$	$P = 2.00 \times 10^5$
Acetate rayon	$P^\circ = 1.58 \times 10^9$	$P = 4.27 \times 10^9$	$P^\circ = 1.58 \times 10^{12}$	$P = 5.13 \times 10^{12}$

* Additional calculations performed since the publication of the first paper in this series [8] have modified slightly some of the values reported there. These modifications in no way alter the conclusions previously drawn.

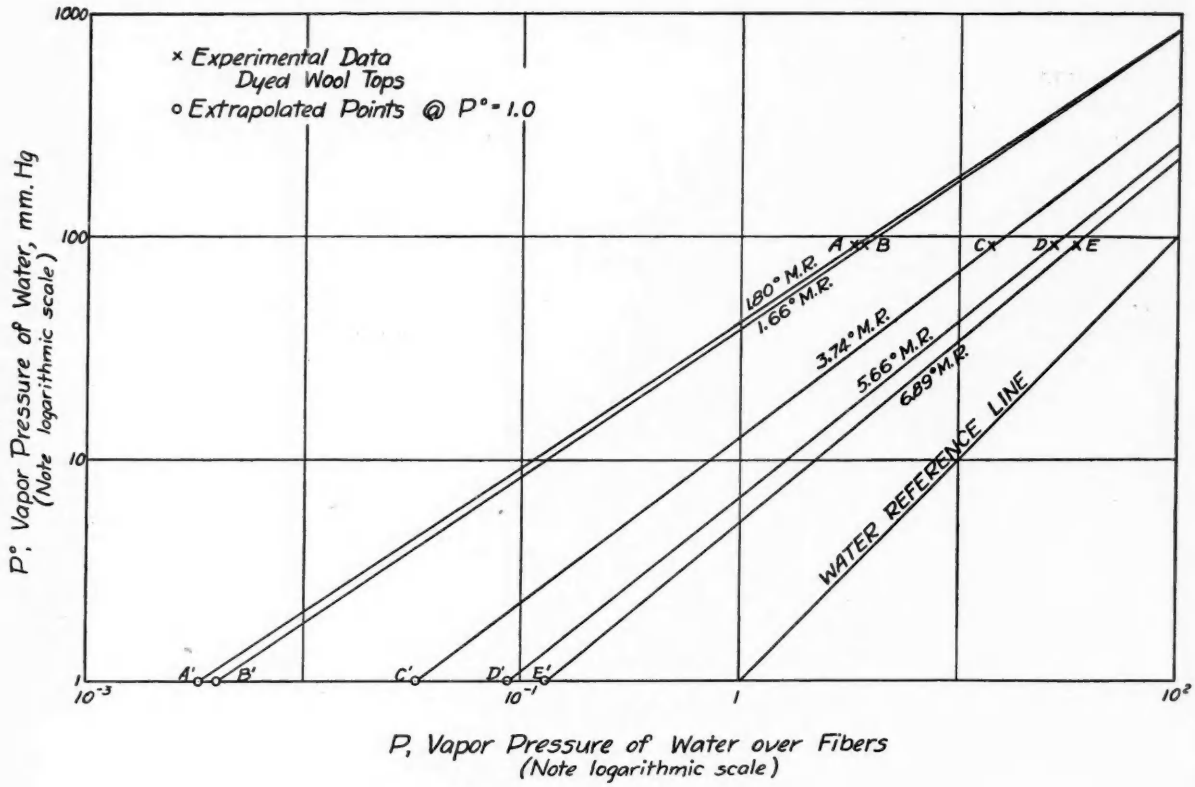

FIG. 10. *Dyed wool tops, Othmer plot.*

regain, and the value so obtained may be placed on the Othmer chart and connected with the common intersection point to give as many isosteres as necessary. By drawing several horizontal lines, each one corresponding to a given temperature (or $P°$), and determining the value of P where each of these constant temperature lines intersects a constant moisture line, one can recalculate the data to the form shown in Figure 12, where moisture regain has been plotted against relative humidity in the usual form. Table III is a summary of the calculations used in this illustration. Also shown in Figure 12 are the original Wiegerink data for carpet wool at the same temperatures for which the data for dyed wool tops are calculated. The wide disparity of regain between the dyed and the relatively pure samples is interesting as an example of the enormous effect of sample history and treatment.

Prototype of Material Not Previously Analyzed

In this case, sufficient experimental evidence must be obtained to determine both the common point and the form of the intercept-regain curve since neither will be available for this material. If information on the swelling property of the fiber when exposed to water is available, it is possible that the location of its common point may be predicted within reasonable limits [8]. In that case, it would only be necessary to indicate the form of the intercept-moisture regain line. Three or four carefully analyzed samples should be sufficient, the only limitation being the

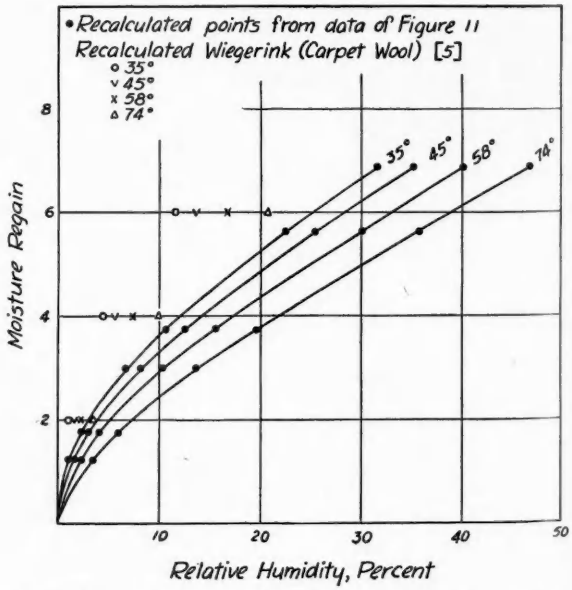

FIG. 12. *Recalculated moisture relations, wools, desorption.*

coverage of a wide set of moisture conditions. The procedure will then be the same as that outlined above for materials already studied.

If the location of the common point cannot be predicted because of complete lack of information on the swelling property of the fiber, experimental points must be taken in greater number and with more careful selection of conditions. In order to obtain the original isotherms on which all succeeding calculations depend, at least four points rather widely separated must be determined at each temperature. It is, of course, preferable to have one or two additional experimental values, but four will do if they are carefully chosen. At least three isotherms must be obtained. Thus, a minimum of 12 experimental values is suggested. After obtaining such data, isothermal lines are drawn on regular regain-relative humidity axes. The procedure is then as outlined in the first paper [8] for the determination of the common point and the recalculation of the data. For simplicity it is suggested that the work be done graphically rather than through the full statistical calculations. It is unlikely that these few data would give any statistical evidence of intersection, and accuracy, consistent with data, may be attained by the graphical solution.

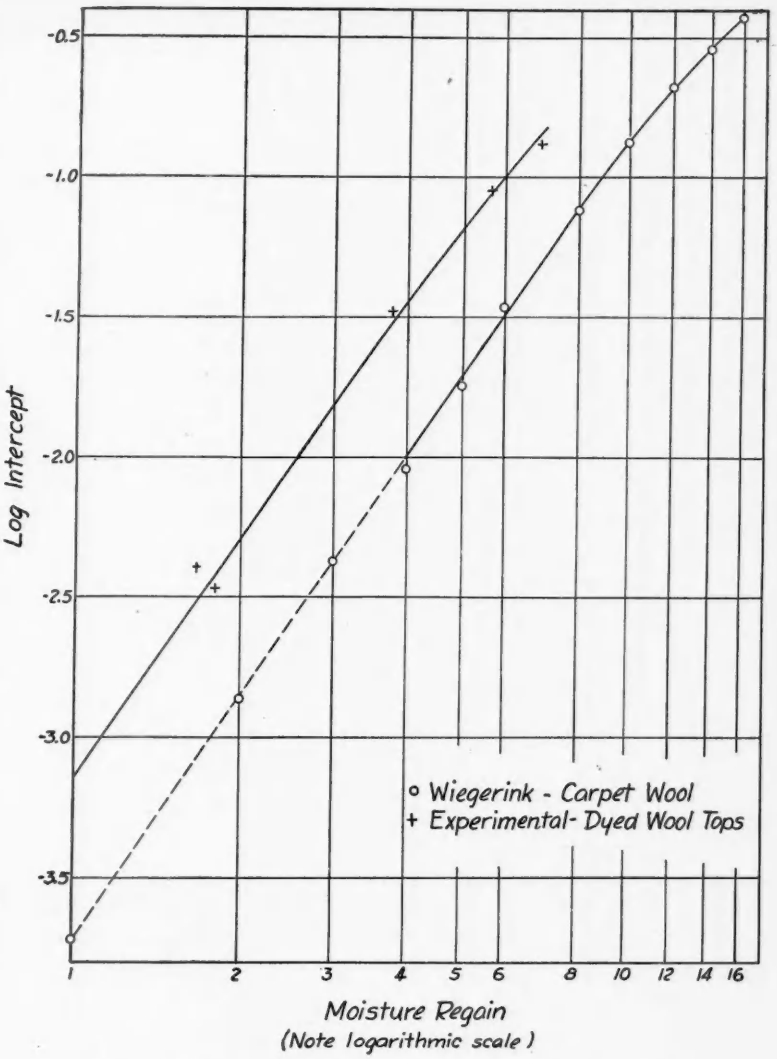

FIG. 11. *Dyed wool tops and Wiegerink carpet wool, intercept curve.*

TABLE III

Sample	MR (%)	P, mm. Hg at $P°$ = 92.5 (experimental)	P, mm. Hg at $P°$ = 1.0 (from Fig. 10)
A	1.80	3.05	0.0034
B	1.66	3.42	0.0040
C	3.74	13.32	0.0334
D	5.66	25.16	0.0899
E	6.89	32.10	0.1314

	t = 35°C ($P°$ = 43.5 mm.)		t = 45°C ($P°$ = 71.9 mm.)		t = 58°C ($P°$ = 138.0 mm.)		t = 74°C ($P°$ = 276.0 mm.)	
MR (%)	Calc. P (mm.)	Calc. RH (%)	Calc. P (mm.)	Calc. RH (%)	Calc. P (mm.)	Calc. RH (%)	Calc. P (mm.)	Calc. RH (%)
1.26	0.520	1.20	1.148	1.60	3.208	2.32	9.624	3.49
1.78	1.051	2.42	2.230	3.10	5.914	4.29	16.800	6.09
3.00	2.888	6.64	5.786	8.05	14.260	10.33	37.400	13.55
3.74	4.652	10.70	9.072	12.62	21.580	15.64	54.540	19.76
5.66	9.844	22.63	18.400	25.59	41.430	30.02	98.720	35.77
6.89	13.740	31.59	25.200	35.05	55.380	40.13	128.600	46.60

It is, of course, theoretically possible to use only four points, two at each of two moisture conditions, to determine the common point of intersection, but because of the variability within samples and in experimental techniques, no reliability could be placed upon the results. Furthermore, additional experimental data would be needed to determine the form of the intercept regain curve.

Summary

The current paper has presented the extrapolation methods actually used in all previous work [2, 5, 8, 9]. In addition, the use of these extrapolation methods in forecasting a complete family of moisture equilibrium curves with very little experimental data has been shown. The Wiegerink data statistically correlated in the previous paper were absolutely necessary for these methods since they represent a consistent set of data for a variety of materials. The essential statistical methods were outlined in the second paper of this series and the theoretical implications resulting from such a correlation were presented in the first. It is hoped that these four papers on moisture equilibrium will be of practical service and will stimulate additional research to clarify the theoretical problems raised.

Acknowledgment

The presentation of these papers would not have been possible without the assistance of many persons. Among them are Dr. A. C. Walker of the Bell Telephone Laboratories, who made available to the authors the original Wiegerink data and who offered many helpful suggestions throughout the course of this study; J. W. Tukey and P. J. McCarthy of the Princeton Mathematics Department, who proposed the mathematical treatment employed; and Carol F. Bowen, who varied the techniques to meet the requirements of the different materials and who performed the extensive calculations required. To all of them the authors express their grateful appreciation.

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ABSTRACTS

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A list of periodicals, the abbreviations used in references to articles, and the addresses of publishers of English language publications is given on page 37 of the January, 1947, issue.

ANALYSIS: TESTING: LABORATORY METHODS

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Leather Fibers

Physical properties of individual leather fibers. I. Evaluation of Young's modulus of elastic extension and percentage permanent set of leather fibers. G. O. Conabere and R. H. Hall. *J. Intern. Soc. Leather Trades Chem.* **30**, 214–27 (1946) (through *Chem. Abstr.* **40**, 6862[6] (Nov. 10, 1946)).

An apparatus is described for measuring extension under load of single fiber bundles teased from leather. Data are given for vegetable-tanned cowhide butt (*I*) and Cr-tanned cowhide belly (*II*). No yield point was found; when stretched to the breaking point the fibers broke in steps, owing to rupture of individual fibrils. When stretched below the breaking point all fibers showed some permanent set (about 20% of total extension); this was not increased on restretching the fiber to the original extension, but increased when the fiber was stretched to a greater extension. Young's modulus ($\times 10^{10}$ dynes per sq. cm.), calculated from the stress-strain curves of fibers previously stretched to eliminate permanent set, was 0.82 to 1.5 for *I* and 0.22 to 0.71 for *II*. The difference between *I* and *II* may be due to the location from which the fibers were taken rather than, or in addition to, the tannage.

Text. Research J. Feb. 1947

Parallel Plate Plastometer

Theory and application of the parallel plate plastometer. G. J. Dienes and H. F. Klemm, *J. Applied Phys.* **17**, 458–71 (June 1946).

An improved parallel plate plastometer for measuring viscous flow in plastic materials is described. Stefan's equation is derived for the case in which the viscous fluid completely fills the space between the plates. The condition wherein plate area is large in comparison to the size of the specimen is also discussed. It is possible, with the aid of the equations given, to calculate the viscosity in poises from plate separation and time measurements. Viscosity-temperature data are presented for polyethylene, and vinyl chloride-acetate resin compounds which show that the log viscosity varies linearly with the reciprocal of the absolute temperature. Data obtained are also in agreement with Flory's relationship: log viscosity varies linearly with the square root of the weight-average molecular weight.

Text. Research J. Feb. 1947 T. J. Dietz

Structure of Polymers

X-rays and the structure of plastic high polymers. G. Champetier. *Peintures, pigments, vernis* **19**, 98–105 (1943) (through *Chem. Abstr.* **40**, 6880[2] (Nov. 10, 1946)).

The x-ray diffraction patterns of high polymers are capable of revealing many details of their structure. Crystalline macromolecules yield patterns of concentric rings. Oriented macromolecules, such as the natural fibers and those obtained by stretching, etc., yield patterns of spots or circular arcs. Amorphous macromolecules show only more or less distinct halos. In the crystalline macromolecules, the measurement of the circular pattern reveals the size of the structural units (—CH_2— in paraffins, —CH_2O— in polyoxymethylenes, —$C_6H_{10}O_5$— in cellulose) and the distance between chains. With fractionated medium-high polymers (higher paraffins; lower polyoxymethylenes) the intensity of the pattern spots allows the estimation of electron density in the molecule and the distinction between covalent (primary) bonds and secondary bonds, and the length of the molecule can be measured from the pattern. In the usual high polymers, which are mixtures of polymers with different molecular weights, this is no longer possible.

However, periods of 1–2 Å. indicate primary valencies, while those of 4–5 Å. indicate secondary valencies between molecules. Amorphous regions within crystalline and oriented macromolecules are revealed by veils or halos in the pattern. Since the diffraction spots become wider and more diffuse as the crystalline regions are reduced, their size can be used to measure the contours of crystalline and amorphous regions in the polymer. A. Guinier (*Thesis*, Paris, 1939) has shown that the dimension and form of amorphous particles can be estimated by measuring the halos they produce around incident monochromatic x-rays. From the diffraction patterns it is possible to follow structural changes accompanying extension (rubber, nylon) and plasticization (nitrocellulose). While it has not been possible to follow the condensation of phenolic resins with x-ray diffraction patterns, Champetier and Boberiether have obtained a series of differing patterns from fractionated urea-CH_2O resin. These, as well as several other patterns, are shown.
Text. Research J. Feb. 1947

Reflectometer

A low-cost reflectometer and method for comparing detergency. Murry L. Hurwitz. *Am. Dyestuff Reptr.* **35**, 83–4 (Feb. 11, 1946).

A diagram is shown of a simple, low-cost apparatus for measuring the 45°-angle reflectance of textile materials used in detergency comparisons. The soiling formula and the procedure for washing, as well as the operation of the apparatus for comparing the results of the detergent action, are given. J. A. Woodruff
Text. Research J. Feb. 1947

Adhesion of Rubber

Coefficients of adhesion of rubber. Pierre Thirion. *Rev. gén. caoutchouc* **23**, 101–6 (1946) (through *Chem. Abstr.* **40**, 6868[2] (Nov. 10, 1946)).

A new apparatus is described whereby a rubberlike material is placed in contact with a smooth glass surface in such a way that tangential internal deformation is avoided. A systematic study of various factors, including pressure, temperature, and speed, showed that elastomers do not conform to the law of Coulomb (*"Théorie des machines simples,"* Paris, 1821); e.g., rubber, Thiokol, and polyvinyl chloride behave like liquids of fixed structure, and their behavior is characterized by a linear relation between the reciprocal of the adhesion coefficient and the contact pressure. The results throw new light on the theory of balance between solid interfaces; e.g., the theory of irreversible molecular cycles to explain the reciprocal friction of rigid solids must be modified to take into account certain thermodynamic factors derived from the kinetic theory of elasticity.
Text. Research J. Feb. 1947

Microscopy of Viscose

The microscopical examination of viscose. Francesco Castellani. *Chimica e industria* **28**, 6–9 (1946) (through *Chem. Abstr.* **40**, 7619[5] (Nov. 20, 1946)).

The microscopic examination of viscose permits detection of (1) amorphous particles of white or yellowish color, transparent, formed in the secondary reactions during the sulfurization (when the degree of sulfurization is high, the number of particles is low), (2) fibers more or less swollen, altered, and not completely dissolved (the rate of filtration depends on the presence of these swollen fibers), (3) crystals or crystalline compounds (Ca Na double carbonate, SiO_2, FeS, etc.) and various other impurities. The microscopic examination is more rapid and useful than the chemical examination.
Text. Research J. Feb. 1947

Viscosity

A photographic-viscometric apparatus and technique. Jesse L. Riley and George W. Seymour. *Ind. Eng. Chem., Anal. Ed.* **18**, 387–90 (June 1946) (through *Bull. Inst. Paper Chem.* **17**, 100 (Oct. 1946)).

An apparatus and procedure for the accurate determination of flow times of dilute solutions in the Ostwald-type viscometer have been developed. Means and measurement of control of the viscometric variables have been designed on the principle of making approximately equal contributions to the final error. A unique feature of the apparatus is the automatic timing device. Motion pictures are made of the meniscus transits in the viscometer and of the virtual image of an accurate, electronically operated clock. Times of transit are interpolated from the pictures to 0.001 sec. Corrected flow times of 3 or 4 check runs for each of the 3 cellulose acetate solutions in dioxane ranging from 0.01 to 0.3% concentration fall within a time range of 0.005%. 14 references.
Text. Research J. Feb. 1947

CHEMICAL AND PHYSICAL RESEARCH

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Cell Wall Deformations

Cell wall deformations in wood fibers. H. E. Dadswell and A. B. Wardrop. *Nature* **158**, 174 (Aug. 1946).

Results of microscopical and chemical study of slip planes and minute compression failures in wood fibers are reported. The authors conclude that a slip plane is a single fold in a cell wall and a minute compression failure is a double fold. They suggest that these cell wall deformations are accompanied by increased micellar surface and intermicellar spaces. It is further concluded that the deformations are morphologically similar to the dislocation marks of textile fibers, and that the explanation in terms of folds in the cell walls is consistent with the optical and mechanical behavior, the staining, and the chemical susceptibility of these cell wall features.
Text. Research J. Feb. 1947 R. W. Eyler

Ageing of Cellulose

Investigations of the ageing of alkali cellulose. K. Lauer, R. Hansen, and E. Franke. *Kolloid-Z.* **108**, 113–20 (Aug., Sept.

1944) (in German) (through *Bull. Inst. Paper Chem.* **17**, 54 (Oct. 1946)).

A study of the ageing of alkali celluloses was made by following kinetically the decrease in D.P. The velocity constant decreased asymptotically during the ageing process and the D.P. of the alkali celluloses approached a limiting value. Molecular degradation products are assumed to cause this retardation. The velocity constants of different cellulosic materials apparently are not correlated with any analytical data of the materials. The heat of activation was determined for a number of cellulosic materials and found to be constant—*i.e.*, approximately 13,340 calories. The difference in the velocity constant must be caused, therefore, by the number of collisions. The importance of the ageing process in the viscose process is emphasized. Degradation during ageing cannot be replaced simply by cooking or bleaching the pulps to a low D.P. The differences appear to be caused by the carboxyl content of the cellulose, which changes during the ageing and finally approximates one carboxyl group per molecule. The changes taking place in the carboxyl molecule were noted and it was evident that some decarboxylation occurred. The filtering properties of the viscose seem improved, provided mild "superoxidation" is allowed to take place during ageing, thus furnishing somewhat more than one carboxyl group per molecule. 12 references.
Text. Research J. Feb. 1947

Chain-length distribution in alkali celluloses ripened by air and by chemical means. H. A. Wannow and Charlotte Feickert. *Kolloid-Z.* **108**, 103–13 (Aug., Sept. 1944) (in German) (through *Bull. Inst. Paper Chem.* **17**, 55 (Oct. 1946)).

A sample of spruce sulfite pulp was converted into alkali cellulose by the usual means. One portion (*I*) was ripened in air to an average D.P. of 266. Another portion (*II*) was ripened to an average D.P. of 296 by the use of hydrogen peroxide. Viscometric measurements were made on both samples; details of the preparations and determinations are given. (*I*) required over 60 hrs. for ripening, whereas (*II*) required less than 10 hrs. When osmometric measurements were made, the average D.P.'s of (*I*) and (*II*) were, respectively, 193 and 216; these differences are discussed. Chain-length distribution studies were made using the cuprammonium and cellulose-nitrate-acetone viscometric methods, as well as the osmometric technique. Results indicated that (*I*) and (*II*) were substantially of the same polymolecular composition and that any differences in chain-length distribution were unimportant. 14 tables, 7 graphs, and 25 references.
Text. Research J. Feb. 1947

Alcoholysis of Cellulose

The alcoholysis of cellulose. Richard E. Reeves, W. M. Schwartz, and J. E. Giddens. *J. Am. Chem. Soc.* **68**, 1383–5 (July 1946).

Cotton fibers were treated with 0.5*N* HCl in methanol, ethanol, methanol-water (9 to 1), and water. Cuprammonium fluidities and copper numbers were run on the products. Alkali solubilities were also determined. Results indicate alcoholysis gives a more rapid degradation than hydrolysis but the upper limit of cuprammonium fluidity is the same in both cases. The alcoholized cellulose is stable to hot-alkali solutions, is nonreducing, and contains measurable amounts of alkoxyl groups attached by glycosidic (acid labile) linkage. Apparently alcoholysis results in the rupture of the glucose-glucose bonds with the addition of a molecule of alcohol. A. R. Macormac
Text. Research J. Feb. 1947

Cellulose Structure

The structure of cellulose. E. Schiebold. *Kolloid-Z.* **108**, 248–65 (Aug., Sept. 1944) (in German) (through *Bull. Inst. Paper Chem.* **17**, 102 (Oct. 1946)).

A critical review is given of all the hitherto-proposed structural formulations for cellulose, beginning with that resulting from the x-ray studies of Sponsler and Dore (*Colloid Symposium Monographs* **4**, 174 (1926)) and ending with the researches of Kubo (cf. *B.I.P.C.* **11**, 229; **13**, 297–8). The structural models, especially those of Meyer and Mark, Meyer and Misch, Andress, and Sauter, are subjected to detailed criticism, based largely on a mathematical re-evaluation of x-ray data. Evidently, the author has in mind a new "model" structure for cellulose which awaits future publication. No new experimental work is included. 97 footnotes.
Text. Research J. Feb. 1947

Structure of Cellulose Fibers

The morphological and the molecular fine structure of fibers, especially of native cellulose fibers. E. Franz, F. H. Müller, and E. Schiebold. *Kolloid-Z.* **108**, 233–48 (Aug., Sept. 1944) (in German) (through *Bull. Inst. Paper Chem.* **17**, 61 (Oct. 1946)).

The technical requirements of a textile fiber are reviewed and summarized. The molecular fine structure essential for a satisfactory fiber is described, and the importance of both amorphous and crystalline regions is emphasized. Morphological structure, however, is as important as is the molecular fine structure. The difference between synthetic and native fibers in this respect is outlined. The morphological "superstructure" of native fibers is the reason for their superiority over the regenerated cellulose fibers. The structure of native cellulose fibers as proposed by Dolmetsch, Franz, and Correns (cf. *B.I.P.C.* **15**, 49–50) is discussed. The theory furnishes an explanation (based on the high-molecular properties of cellulose) for the great strength of fibers, despite their subdivision into small building units (dermatosomes). 80 footnotes.
Text. Research J. Feb. 1947

Viscosity of Cellulose

Further investigations of cellulose in solution. H. Erbring. *Kolloid-Z.* **108**, 16–20 (July 1944) (in German) (through *Bull. Inst. Paper Chem.* **17**, 53 (Oct. 1946)).

Even today it is not clear how molecules of cellulose and cellulose

derivatives are dispersed in concentrated solutions. A number of experimental data seem to support the theory of a nonmolecular dispersion of the chain molecules. A review of previous work in this field is given. Founded on Sakurada's investigations (*Kolloid-Z.* **61**, 50 (1932)), the viscosities of acetyl- and benzylcellulose were measured in different solvent-nonsolvent mixtures. In one series the derivatives were dissolved in different organic solvents, varying amounts of a nonsolvent (methyl alcohol) were added and the viscosity was measured. In another series the derivatives were dissolved directly in mixtures similar to those described above and their viscosities were measured. The viscosities were always higher in the second case. Several graphs are given to show this difference over a wide range of solvent-nonsolvent ratios. The higher the concentration of the nonsolvent, the greater was this difference. A possible explanation for this behavior is given. When the chain molecules are dissolved directly in a solvent-nonsolvent mixture, association between molecules takes place during solution. If the cellulosic materials are first dissolved in the pure solvent, a long time-period elapses before the same noticeable association takes place, thus accounting for a difference in the viscosities.

Text. Research J. Feb. 1947

Viscosity of Nitrocellulose

A note on the viscosities of solutions of nitrocotton in a mixed nitroglycerine-nitroglycol solvent. D. Fensom. *Can. J. Research* **24B**, 83–7 (May 1946).

Viscosity-concentration data are presented for a number of nitrocellulose samples (11.81–12.26%N) in 80:20 nitroglycerine-nitroglycol. The data follow the Arrhenius equation within experimental error, but the plot of log relative viscosity vs. concentration does not pass through the origin. Intrinsic viscosity appears to be considerably higher in the mixed solvent used in this work than in acetone. A few data on the heat-stability of the solutions are given. W. E. Davis

Text. Research J. Feb. 1947

Structure of Immature Cotton Fibers

Structure and plasticity of undried cotton fibers. Earl E. Berkley and Thomas Kerr. *Ind. Eng. Chem.* **38**, 304–9 (Mar. 1946).

X-ray patterns were made of cotton fibers taken from the boll about 23 days after flowering (4 to 5 days after secondary thickening started) and at other stages until after the boll opened. Practically no evidence of crystallinity appeared until the fiber started to dry. The undried fibers were birefringent and possessed a fibrillar structure. This would indicate not only an orientation of the cellulose chains but also the presence of a large number of water molecules separating the chains. The pattern of native cellulose appears as soon as the fiber starts to dry. Crystallization was also caused by stretching of the undried fibers which were plastic or by replacement of the water with a nonpolar solvent. Stretching also increased the slope of the cellulose spiral and the fiber strength. Fibers impregnated with glucose and dried without tension showed diffraction lines of cellulose and glucose oriented along the fiber axis. Undried flax fibers showed slight signs of crystallinity. 4 photomicrographs and 15 x-ray patterns are given. A. R. Macormac

Text. Research J. Feb. 1947

Jute

Effect of dyeing, mercerizing, and intensively delignifying jute fibers on their structure. N. G. Banerjee, B. S. Basak, and R. K. Sen. *Nature* **158**, 100 (July 1946).

X-ray diffraction patterns for raw jute after dyeing with Congo red, after delignification, after mercerization and dyeing with methylene blue, and after delignification and dyeing with methylene blue are presented and discussed. It is concluded that intensive delignification does not affect the lattice structure and size of the cellulose crystallites, but that the ordering along the fiber axes has been caused to deteriorate considerably. It is further shown that the process of dyeing does not affect the internal structure or alignment of crystallites of raw, delignified, or mercerized cellulose.

Text. Research J. Feb. 1947 R. W. Eyler

Solutions of Macromolecules

The behavior of macromolecules in inhomogeneous flow. H. A. Kramers. *J. Chem. Phys.* **14**, 415 (July 1946).

Following up the ideas of W. Kuhn's skein theory, J. J. Hermans has recently elaborated a theory of the properties of dilute solutions of highly polymerized molecules. Hermans describes the behavior of linear molecules by the diffusion of their end-points. He finds that the contribution of a single molecule to the viscosity coefficient as well as to the double refraction of the solution is proportional to the square of the degree of polymerization, in agreement with the experiments. In the present article we investigate the statistical behavior of the individual links of the solved molecules. In this way we arrive at results equivalent with those of Hermans. Our method, however, can also be applied to molecules which possess branching points and rings.

Text. Research J. Feb. 1947 Author

Instability of Polymer Solutions

Instability of solutions of high polymers. J. A. Morrison, J. M. Holmes, and R. McIntosh. *Can. J. Research* **24B**, 179–91 (Sept. 1946).

This investigation was undertaken because of the necessity for protracted storage in iron containers of solutions of polymers in technical solvents. It consisted in determination of viscosity changes in solutions of polyvinyl acetate, polystyrene, and polymethyl methacrylate in nitrobenzene, bis(2-chloroethyl) ether, and bis (2-chloroethyl)sulfide, on ageing at 60°C in the presence of varying amounts of $FeCl_3$, $FeCl_2$, and air. Intrinsic viscosity determinations were carried out on the polymers recovered from the aged solutions, and in some cases the aged solutions were examined for the presence of structural viscosity. In

general, viscosity changes in the aged solutions were paralleled by changes in intrinsic viscosity of the recovered polymers, indicating that actual changes in molecular weight were probably taking place. In the presence of air alone, the viscosity always dropped, while $FeCl_3$ alone caused an increase in viscosity which ultimately resulted in gel formation. $FeCl_2$ alone had practically no effect. When both $FeCl_3$ and air were present, the viscosity first dropped, then increased again until a gel was formed. The viscosity at the point of gel formation was frequently less than that of the unaged solution, indicating the essential independence of the degradation and gel-forming reactions. $FeCl_2$ in the presence of air behaved much like $FeCl_3$. The effect of a given amount of impurity was dependent upon the particular solvent used. Variations in k, the slope constant in the viscosity-concentration relation, during ageing were also noted, especially with $FeCl_3$ present, which is consistent with a change in structure of the polymer in the presence of iron.

Text. Research J. Feb. 1947 W. E. Davis

Molecular Weight of Polyvinyl Acetate

The molecular weight of some polyvinyl acetates. R. McIntosh and J. A. Morrison. *Can. J. Research* **24B**, 192–9 (Sept. 1946).

The molecular weights of 2 series of polyvinyl acetates, 1 series aged in solution in the presence of air, the other in solution in the presence of $FeCl_3$, were determined in the osmometer. It was found in this way that the changes in viscosity which occur during such ageing (*Can. J. Research* **24B**, 179–91 (Sept. 1946)) are due, in part, at least, to changes in molecular weight. The relationship between intrinsic viscosity and number-average molecular weight is of the same form for unaged polyvinyl acetate and for the 2 series of aged samples, but the constants in the equation $[\eta]=KM_n^a$ are different in the 3 cases. On the assumption that all 3 types of material follow the logarithmic distribution law of Lansing and Kraemer (*J. Am. Chem. Soc.* **57**, 1369–77 (1935)), non-uniformity coefficients were calculated for the aged samples. The change in the coefficient with ageing time was quite small. W. E. Davis

Text. Research J. Feb. 1947

Membranes for Osmotic Pressure Measurements

The influence of membrane preparation on the osmotic pressure of polyvinyl acetate in acetone. R. E. Robertson, R. McIntosh, and W. E. Grummitt. *Can. J. Research* **24B**, 150–66 (July 1946).

Two types of membranes were studied, both prepared from uncoated cellophane from which plasticizer had been removed by soaking in water. One type (designated Carter and Record) was prepared by soaking in ethanol-water mixtures of various compositions (high-ethanol mixtures giving membranes of low permeability and vice versa), then in absolute ethanol for 12 hrs., finally in anhydrous acetone (the solvent used in the osmometer). The other type was prepared by soaking in NaOH solutions of varying composition (permeability increases with NaOH concentration), dipping in water or 1:1 ethanol-water, soaking in anhydrous ethanol for 15 min., then in anhydrous acetone. With the Carter and Record membranes, high permeability to solvent was usually accompanied by permeability to the low-molecular-weight portion of the solute, while the caustic-treated membranes showed no evidence of permeation by solute, although they passed solvent very rapidly. Another difficulty with the Carter and Record membranes was traced to adsorption of solute by the cellophane, which caused a considerable lag in attainment of equilibrium osmotic pressure, and, in cells of sufficiently high ratio of membrane area to cell volume, changed the solute concentration appreciably, so that reliable values of osmotic pressure could be obtained only by pouring out the equilibrium solution and refilling the cell with fresh solution. No adsorption difficulties were encountered with the caustic-treated membranes as long as they were not washed so much as to remove all the caustic. The Carter and Record membranes, even when the cell was refilled to eliminate the adsorption effect, did not give the same value of π/c at zero concentration as the caustic-treated membranes, nor was the slope of the π/c vs. c lines the same. The differences could not be accounted for on the basis of differences in permeability of the membranes to low-molecular-weight material. The behavior recorded in this paper will probably not be fully explained until the mechanism of membrane action is better understood than at present. The paper also presents data relating intrinsic viscosity to number-average molecular weight for a series of polyvinyl acetates. W. E. Davis

Text. Research J. Feb. 1947

Birefringence of Synthetic Rubbers

The nature of the dynamic double-refraction of rubber solutions and the shape of rubber molecules. V. Tsvetkov and A. Petrova. *Rubber Chem. Tech.* **19**, 360–84 (Apr. 1946).

Values are given for birefringence and angle of extinction as functions of velocity gradient for a number of synthetic rubbers, and a method of correcting the birefringence for concentration effects is worked out. It is shown that particle shape and anisotropy contribute very little to the birefringence, which is influenced primarily by the orientation due to flow. A study of birefringence induced by an electric field was also made, and data are given on the relation of axial ratio to degree of polymerization for a number of samples. The theories involved in some of the measurements are discussed at some length.

Text. Research J. Feb. 1947 W. E. Davis

Structure of Wood Fibers

The fine structure of wood fibers. H. Dolmetsch. *Kolloid-Z.* **108**, 183–92, 4 plates (Aug., Sept 1944) (in German); cf. *B.I.P.C.* **15**, 49–50; **16**, 135 (through *Bull.*

decrease in strength of regenerated fibers and the increase in strength of native fibers on wetting. The rupture of regenerated fibers is not caused by slippage or shearing of the cellulose chains but by the actual rupture of the cellulose chains themselves. The molecular net structure is very irregular in a regenerated fiber and causes an uneven stress distribution over the cross section of the fiber, thus producing a simultaneous break of a few cellulose chains. In the dry state, however, there are several points of adhesion (*Haftpunkte*) between the cellulose chains, giving a more uniform stress distribution and a higher fiber strength. On the other hand, in the natural fibers the cellulose chains are arranged far more regularly. The structure is compared with that of a cable. This is especially true in the wet state, when the intermolecular forces are small, permitting the chains to assume the most uniform stress distribution, when wet, and thereby imparting an augmented wet strength to the fiber. 11 footnotes.
Text. Research J. Feb. 1947

BLEACHING: DYEING: FINISHING

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Effect of Mercerization on Dyeing

Some aspects of color. R. W. Jacoby. *Am. Dyestuff Reptr.* **35**, P56–8 (Jan. 28, 1946).

Experiments have shown that the obviously greater depth of shade of mercerized as compared to unmercerized cotton when dyed under identical conditions is due to (1) a slight increase in the amount of dye taken up, (2) a material change in the optical properties whereby reflectance is reduced, and (3) a considerably smaller amount of penetration of dye into the fiber. The difference of depth obtained when bright filament rayon and cotton are dyed under the same conditions is similarly explainable. Many finishing treatments change the optical properties of dyed pieces and cause difficulties when matching to standards. Wet goods are shown to have only about ½ the spectrophotometric brightness of the same goods dry. K. S. Campbell
Text. Research J. Feb. 1947

Effect of Vat Dyes on Cotton

Vat dyes that do not weaken fibers on insolation. V. A. Blinov. *Tekstil. Prom.* **5**, No. 11/12, 47–9 (1945) (through *Chem. Abstr.* **40**, 5567[6] (Sept. 20, 1946)).

Dyed specimens of cotton and silk were exposed to the sun for 42 days, directly or under a glass cover. Fibers from the insolated fabrics were then tested for their tensile strength. Cotton exposed under glass lost an average of 5% of its strength; cotton exposed directly lost an average of 25%. The corresponding values for silk were 39 and 55.6%. Vat dyes containing N in a ring which contains also carbonyl groups were least deleterious. Such a dye was flavanthrene. The presence of a triazine ring did not prevent the destructive effect of the dye on the fiber. Condensation products of dicyanodiamide and CH_2O retarded the effect of insolation on undyed specimens, but on vat-dyed specimens their retarding effect was very slight.
Text. Research J. Feb. 1947

Black Shades on Rayon

The all-important question of good black shades: dyes and methods for acetate and viscose rayons. Anon. *Silk and Rayon* **20**, 980, 982 (Aug. 1946).

A review is given which describes the uses of oxidation blacks and the logwood-mordant process. Typical dyeing formulas are presented.
Text. Research J. Feb. 1947 J. F. Keating

Screen Printing Defects

Faults in rayon screen printing. Anon. *Rayon Text. Mo.* **27**, 428–32 (Aug. 1946).

Information pertinent to the prevention and cure of faults in rayon screen printing is given. Marking off, discharge printing troubles, color unevenness, sparking, bleeding, halos, flushing, and penetration difficulties are discussed.
Text. Research J. Feb. 1947 J. F. Keating

Desizing

Use of enzymes in preparing fabrics for dyeing. Anon. *Silk and Rayon* **20**, 1112–17 (Sept. 1946).

Two methods for evaluating the efficiency of desizing enzymes are discussed critically. Measurement of the change in viscosity of a starch solution as a function of enzyme concentration is preferred to Lenk's method of following the starch conversion, using iodine as an indicator, to the disappearance of amylopectin.
Text. Research J. Feb. 1947 J. F. Keating

Flameproofing

Durable flameproofing suitable for cotton outer garments. K. S. Campbell and J. E. Sands. *Textile World* **96**, 118, 119, 222, 226 (Apr. 1946).

The following formula is proposed for flameproofing 2.5-yds./lb. cotton drill to meet the requirements of Fed. Spec. CCC-D-746 and J.Q.D. Spec. 242:

	Parts by weight
(A) Urea-formaldehyde resin monomer	9.62
Catalyst for resin	0.40
Water	22.26
Chlorinated paraffin (70% chlorine content)	14.40
Chlorinated paraffin (42.5% chlorine content)	12.00
(B) Solvent (hydrocarbon; *e.g.*, Stoddard solvent)	25.15
Oil-modified alkyd resin	3.89
(C) Antimony oxide, 300 mesh or finer	12.28

The solids in (A) and (B) are dissolved in the indicated solvents; then (A) is added to (B) with stirring and forms an emulsion into which the antimony oxide then is dispersed. H. J. Burnham
Text. Research J. Feb. 1947

Regeneration of Mercerizing Solutions

Regeneration and evaporation of sodium hydroxide solutions after

Inst. Paper Chem. **17,** 61 (Oct. 1946)).

A morphological discussion is presented dealing with the structure of the cell wall of spruce-wood and with an examination of partially delignified spruce pulps. The various "layers" present in spruce tracheids, their orientation, location in the cell wall, their relative number per cell, their average dimensions, and their (possible) chemical composition are discussed at length, illustrated by means of photomicrographs and schematic drawings, and tabulated. The structure of the secondary cell wall follows from a study of these more or less sharply defined "layers." Of special interest is the fact that the surface, transversely placed spirals become visible even in untreated tracheids of spruce summerwood, and that certain cleavage layers (*Trennflächen*) of the flat spirals become soluble in organic solvents after nitration of a wood section, whereas the actual spiral layers themselves, because of their high lignin content, remain insoluble. Examination of slightly delignified pulps made possible a thorough examination of the primary cell membrane and its structural "element" rich in lignin. The action of various chemicals on the cell wall is described briefly. 15 references.

Text. Research J. Feb. 1947

Elastic Properties of Tire Cords

Hysteresis and related elastic properties of tire cords. Helmut Wakman and Edith Honold. *J. Applied Phys.* **17,** 698–711 (Aug. 1946).

When specimens of representative tire cords are stressed repeatedly under cyclically applied loads, the hysteresis effect, elastic modulus, elongation and rate of growth may be shown to be related to the number of cycles, load, moisture content, and temperature. Under repeated stressing at a rate of 1 cycle per sec. such cords as rayon, cotton, and nylon exhibit a reduction in hysteresis loss as well as an increase in elastic modulus and elongation. In general, an increase in moisture content of the cord is accompanied by an increase in hysteresis and elongation, and a reduction in elastic modulus. However, cotton cords appear to exhibit an initial increase in elastic modulus with increase in moisture, the maximum occurring at about 4 to 5% moisture content. Tire cords vary in the effect of temperature on their hysteresis behavior and modulus. Nylon exhibits an increase in hysteresis and a drop in modulus with increase in temperature from 25°C to 145°C. The relationship between temperature and hysteresis, or temperature and modulus, for rayon and cotton is more complex. T. J. Dietz

Text. Research J. Feb. 1947

Fiber Strength and Structural Relationships

Relationship between bending endurance (Knickbruchfestigkeit) and the spiral structure of cellulose fibers. P. H. Hermans. *Kolloid-Z.* **108,** 180–2 (Aug., Sept. 1944) (in German); cf. *B.I.P.C.* **15,** 90–1 (through *Bull. Inst. Paper Chem.* **17,** 63 (Oct. 1946)).

Experiments were carried out with "model," carefully prepared viscose fibers which varied in the orientation of their cellulose chains. Thus, under identical conditions of preliminary stretching and under carefully controlled conditions of relative humidity, temperature, load, and rate of bending (carried out in Hennig's bending tester (cf. *Melliand Textilber.* **17,** 121–3 (1936)), the bending endurance increased significantly with the fineness of the thread up to 40% elongation (prior to the test); past this point, it dropped quite rapidly, and, when the elongation had reached 120%, it was invariably very low. In general, the better the orientation, the lower was the bending endurance of nontwisted threads. The excellent bending endurance of natural, well-oriented cotton fibers is ascribed to their spiral structure rather than to orientation, because the highly oriented ramies show a certain weakness in bending endurance. Freshly prepared isotropic cellulose threads (initial degree of swelling 5.5) were progressively stretched while still swollen. One set (*I*) of such fibers was dried and another set (*II*) was twisted while the length of the thread was kept constant during torsion. In either set, knots were tied in one group of the fibers (*Ia*) and (*IIa*) whereas another set (*Ib*) and (*IIb*) was kept free from knots. In all cases, the tensile strength and elongation to the point of rupture were noted. In the case of (*Ib*), the tensile strength increased progressively with elongation (*i.e.*, orientation). In the case of (*Ia*), it passed through a maximum (at 50% elongation). In either case, elongation to the point of rupture naturally decreased with progressive elongation. In the case of (*IIb*), there was no significant change in tensile strength over that of (*Ib*). However, in (*IIa*) there appeared to be no maximum through which the tensile strength changed with elongation. Furthermore, the tensile strength for corresponding elongation was always significantly higher in the case of (*IIa*) than of (*Ia*). Thus, it is clearly shown that torsion favorably influences the strength of threads that carry knots. (This tensile strength in knotted threads is termed "relative *Knotenfestigkeit*" (K.F.).) The greater the number of twists (other variables being kept constant), the higher was the K.F. Another conclusion reached from the experimental data is that the brittleness of a fiber is lowered by torsion and, if it were possible to twist artificial fibers while they are still in the swollen state, their properties would be greatly improved. (The practicability of such a procedure, however, is questioned by the author.) The optimum number of twists per cm. is inversely proportional to the diameter, and mathematical relationships have been developed whereby this may be calculated.

Text. Research J. Feb. 1947

Wet Strength

The wet strength of regenerated cellulose fibers. P. H. Hermans. *Kolloid-Z.* **108,** 177–9 (Aug., Sept. 1944) (in German) (through *Bull. Inst. Paper Chem.* **17,** 62 (Oct. 1946)).

An explanation is given for the

has influence on the condensation. High content of dry matter, as in British gum, gives best results. Good steam regulation is essential. Undesirable reactions may occur in the time interval between printing and steaming. Aniline black produces acids upon steaming; they are rendered harmless by the use of Kollamine. If rapid dyes are used, acetic acid and formic acid in the ratio 3:1 have to be blown in together with the steam.

Text. Research J. Feb. 1947

Water-Resistant Treatments

Water-resistant treatments. Raymond A. Pingree. *Am. Dyestuff Reptr.* **35**, 124–7 (Mar. 11, 1946).

An O.C.Q.M. textile team paper describing the efforts in Germany in trying to lower the water imbibition of viscose rayon and to promote water-repellency of viscose fabrics. The chemical formulas and methods of application were given for Persistol VS, KF, compounds known as Ho 1-105, Ho 1-107, Ho 1-193, and Kaurit AK-2, F-1.

J. A. Woodruff

Text. Research J. Feb. 1947

Chlorination of Wool

Wet chlorination of wool textiles and mixtures. W. A. Edwards. *Rayon Text. Mo.* **27,** 479–82 (Sept. 1946).

To produce a shrink-resistant finish on wool by the improved "Negafel" process, the fabric, preferably dry, is treated with a solution of hypochlorite, CO_2, and formic acid below 18°C. Pretreatment with alkaline scouring solutions must be avoided since subsequent chlorination then produces fiber degradation. When prewetting is necessary, *e.g.*, with tightly knitted goods, the wetting liquor should contain a nonalkaline wetting agent and should be held to pH 5.0–6.0. J. F. Keating

Text. Research J. Feb. 1947

Unshrinkable Wool

The use of polymers to make wool unshrinkable. Part I. Anhydrocarboxyglycine. A. W. Baldwin, T. Barr, and J. W. Speakman. *J. Soc. Dyers and Colourists* **62**, 4–9 (Jan. 1946).

The reaction between anhydrocarboxyglycine and wool to build up on the reactive side chains of the wool what is described as essentially a protein film is shown to control the shrinkage of wool flannel, apparently by masking the scales of the wool. The optimum conditions of treatment, the properties of the wool so treated, and the reaction between the wool and the anhydrocarboxyglycine are discussed.

J. A. Woodruff

Text. Research J. Feb. 1947

Textile Finishes

Facts and fiction about fibers and finishes. Raymond B. Seymour. *Am. Dyestuff Reptr.* **35**, 128–30 (Mar. 11, 1946).

A brief discussion of some of the characteristics of textile fibers as well as short descriptions of various modern textile finishes are given.

J. A. Woodruff

Text. Research J. Feb. 1947

FIBERS: YARNS: FABRICS: MECHANICAL PROCESSES

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Cell Structure

Elasticity, plasticity and fine structure of plant cell walls. Otto Treitel. *J. Colloid Sci.* **1**, 327–70 (July 1946).

From measurements of the mechanical behavior (elasticity and plasticity) of cylindrical plant tissues in comparison with materials of known structure conclusions regarding the submicroscopic structure are drawn. The mechanism of the physical growth of the primary cell wall is explained by passage of glucose units from protoplasm to the meshes of the cellulose network in the primary wall. It is concluded that the submicroscopic structure of plant tissues generally is not changed by the influences of humidity, temperature or tension. E. D. Klug

Text. Research J. Feb. 1947

Processing Flax

The processing of domestic flax for textile use. I. Decortication. J. L. Taylor. *Georgia School Technol., State Eng. Expt. Sta. Bull.* No. 9, 28 pp. (1946) (through *Chem. Abstr.* **40**, 5568[7] (Sept. 20, 1946)).

A report of experiments made to produce shive-free flax fibers. Indications are that the best system of machines consists of (1) a stapling machine, (2) retting tanks, (3) a tunnel drier, (4) a decorticator, (5) a dusting machine, and (6) a fiber condenser. 2 and 3 are not discussed; the other are. The quality of fiber produced is not sufficiently high to permit the economical spinning of 100% flax yarn on cotton machinery. It is satisfactory for use in blends with other fibers. **II. Retting and degumming.** *Ibid.* No. 10, 24 pp. A report of experiments made. Flax straw is stapled (2–3 in.), then retted 5 days in tap H_2O at 85–95°F. For degumming, a solution of either 4% NaOH, or 1% Na_2CO_3 and 2–5% Na_2S, at 330–340°F, for 1–1¾ hrs., at 90–105 lbs. pressure, gives fiber satisfactory for processing and spinning on cotton textile machinery.

Text. Research J. Feb. 1947

Hydrated Jute Fiber

Hydrated cellulose from jute fiber. S. C. Serkar and N. N. Saha. *Nature* **157**, 839 (June 1946).

Unstretched raw jute fiber which has been treated with 30% sodium hydroxide solution, washed with water, and dried in free air at room temperature for 3 days gives an x-ray diffraction pattern which is different from that due either to ordinary hydrated cellulose or to the "water cellulose" reported by Sakurada and Hutino. The physical properties of this hydrated cellulose from jute are reported to be similar to those of coarse wool and superior to those of hydrated cellulose obtained from jute fiber with 12% sodium hydroxide.

Text. Research J. Feb. 1947 R. W. Eyler

mercerization. B. M. Cherkinskiĭ. *Tekstil. Prom.* **6**, No. 7/8, 34–7 (1946) (through *Chem. Abstr.* **40**, 7642[8] (Nov. 20, 1946)).

An installation for regenerating spent mercerizing solutions is described. The spent solution containing Na_2CO_3 is regenerated with lime in a causticizer and is then concentrated in evaporators.

Text. Research J. Feb. 1947

Mildewproofing Agents

Fungicidal activity of bisphenols as mildew preventives on cotton fabric. Paul B. Marsh and Mary L. Butler. *Ind. Eng. Chem.* **38**, 701–5 (July 1946).

Tests made on cotton duck containing various bisphenols by inoculation with *Chaetomium globosum*, *Metarrhizium glutinosum*, *Aspergillus niger*, or soil suspensions and by soil-burial tests indicate that 2,2′methylenebis (4-chlorophenol), commercially known as G-4, is the best of compounds of this type. The corresponding bromine compound was as good but no better. Additional chlorines on the ring decreased the fungistatic effectiveness. So did the substitution of alkyl group on the phenolic group. The substitution of nitro groups for chlorine eliminated the fungistatic properties. Compounds containing several *p*-chlorophenol or *p*-cresol rings connected by a methylene group were ineffective.

A. R. Macormac

Text. Research J. Feb. 1947

Resin Finishes

Some new developments in resin finishing. C. S. Jones. *Fibres, Fabrics & Cordage* **13**, 290–1 (1946) (through *Chem. Abstr.* **40**, 6822[3] (Nov. 10, 1946)).

The discussion deals with the use of thermosetting and thermoplastic resins in the textile industry, fire-resistance of fabrics, and cellulose ether finishes.

Text. Research J. Feb. 1947

Resin Finishing

Practical experiences and critical evaluation of the swelling-proof finishing of regenerated cellulose. Hugo Bartl. *Allgem. Textil-Z.* **2**, 212–14 (1944) (through *Chem. Abstr.* **40**, 5927[6] (Oct. 10, 1946)).

Methods of swelling-proof finishing by means of the incorporation of synthetic resins and chemical treatment of cellulose with CH_2O are discussed and evaluated. There is the danger that the properties of some textiles may not be improved but may be impaired badly by unsuitable swelling-proof treatment.

Text. Research J. Feb. 1947

Treatment of Fibers with Resin

Coating textile fibers with resins. A. I. Matetskiĭ. *Legkaya Prom.* **1945**, No. 10/11, 28–9 (through *Chem. Abstr.* **40**, 7643[7] (Nov. 20, 1946)).

The fiber is saturated with a catalyst and treated with a mixture of steam and vapors of a monomer that can be polymerized within the fiber under these temperature conditions. Waste Plexiglass was distributed to give approximately 80% of a highly volatile, colorless liquid, supposed to be a mixture of acrylic and methacrylic acids and their methyl and ethyl esters. $(NH_4)_2S_2O_8$ or $(C_6H_5CO)_2O$ was used as catalyst. Wool, cotton, natural silk, viscose yarn, fabrics, felt, and tricot were coated by this method.

Text. Research J. Feb. 1947

Rotproofing

Service test of copper-treated cotton sandbags. J. D. Dean, W. B. Strickland, and W. N. Berard. *Am. Dyestuff Reptr.* **35**, 346–8 (July 15, 1946).

Tests conducted in New Orleans over a period of 19 mos. showed the relative value of 8 different copper-containing rotproofing treatments in prolonging the service life of cotton sandbags. Concentrations applied were in accordance with recommended practice. 40 bags were set out, including 5 replicates of each treatment, in 4 uniform lots in March, June, September, and December; the bags contained clean sand and were laid out singly on fertile soil. Results obtained were in good general agreement with the results of the accelerated soil-burial method which had indicated the creosote treatments (copper naphthenate–creosote, and cuprammonium–creosote) to be definitely superior to copper naphthenate alone in rot-resistance, the cuprammonium carbonate to be its equal, and the others to be inferior.

K. S. Campbell

Text. Research J. Feb. 1947

Slashing

Cotton warp sizing. Dr. Paul V. Seydel. *Cotton* **110**, 63–6, 69 (Aug. 1946).

The first paper of a series. A comprehensive picture of the sizing operation is given including various factors which influence the choice of a so-called "ideal" size and the action and importance of sizing.

H. J. Burnham

Text. Research J. Feb. 1947

Steaming Printed Fabrics

The steam treatment of printed fabrics. J. van de Voorde. *Textielwezen* **2**, 255–7 (1946) (through *Chem. Abstr.* **40**, 5924[8] (Oct. 10, 1946)).

The treatment of fabrics with saturated steam at 102–104° for 2 to 120 min. immediately after printing serves to fix the dye, to coagulate thickeners like albumin, to develop dyes like aniline black, and to dissolve the leuco form of vat dyes. The steam has to be used at slightly increased pressure to avoid entrance of air into the apparatus. The composition of the printing paste also influences the result of the steam treatment of vat dye; K_2CO_3 and Rongalit C condense steam, the dissolving of K_2CO_3 gives off heat, while the dissolving of Rongalit absorbs it. Overheating in a reducing nonacid medium is less harmful than in oxidizing solution. The decomposition of Rongalit which begins at 85° is endothermic while the reduction of the vat dye by Rongalit is strongly exothermic. The condensed water can extract salts of the thickener. The amount of dry matter in the paste also

Kapok and Bubblfil

Kapok and its substitute, Bubblfil. C. S. Jones. *Indian Text. J.* **66**, 436–8 (Feb. 1946).

A review of the origin, properties, and uses of kapok and "Bubblfil."

Text. Research J. Feb. 1947 R. K. Worner

Milkweed-Stem Fiber

Fiber from the stems of common and swamp milkweeds. E. G. Nelson and S. T. Dexter. *Mich. Agr. Expt. Sta. Quart. Bull.* **28**, 2–7 (1945) (through *Chem. Abstr.* **40**, 6821[1] (Nov. 10, 1946)).

Fiber was obtained from *Asclepias syriaca* and *A. incarnata*. It was retted, dried, and cleaned. Based on the yield and quality of the fiber, it cannot compete with other fibers which can be produced in Michigan.

Text. Research J. Feb. 1947

Plastics

The development of plastics for peacetime textiles. D. H. Powers. *Am. Dyestuff Reptr.* **35**, 76–8 (Feb. 11, 1946).

A review of the more recent uses of plastics as films, in impregnation, as sizing materials, as binders, and as fibers. J. A. Woodruff

Text. Research J. Feb. 1947

German Plastic Fibers

The fabrication of plastics. Extracts from report by members of the Combined Intelligence Objectives Subcommittee. *Silk J. Rayon World* **22**, 37–9 (May 1946).

Methods used by the I. G. Farbenindustrie at Wolfen for spinning plastic fibers and yarns are described. R. K. Worner

Text. Research J. Feb. 1947

Acetate Rayon Developments

Acetate rayon. Harold DeWitt Smith. *Am. Dyestuff Reptr.* **35**, P71–2 (Feb. 11, 1946).

The wartime uses of acetate rayon dictated by its specific properties of thermoplasticity, electrical resistivity, affinity for fluorescent dyes, and mildew-resistance are discussed. Acetate rayons of very high strength and low elongation, and with normal strength and very high elongation, were produced for several types of parachute uses. Progress in combating fume fading of dyed acetate, and in technique of dyeing is reviewed. K. S. Campbell

Text. Research J. Feb. 1947

New Rayon Developments

Developments in viscose rayon. F. Bonnet. *Am. Dyestuff Reptr.* **35**, P68–70 (Feb. 11, 1946).

The production of rayon (724,000,000 lbs. of all types in 1944) has almost doubled in the last 6 years, a large factor in this increase being due to the government program calling for 240,000,000 lbs. of viscose rayon tire yarn. This expansion program for war production has put the industry ahead by about 10 years; information gained has made it possible to produce "semi-strong" yarns of suitable extensibility and of very fine filament (1 den.), and many new and attractive fabrics with unusual properties are predicted. Although this stronger type of viscose rayon has a much higher wet strength and a smaller proportional wet-strength loss than regular viscose, there is but little difference in moisture absorption between the two. Treatments which will greatly improve the resistance to laundering of rayon fabrics are in prospect. A number of new, minor uses for viscose staple are described. K. S. Campbell

Text. Research J. Feb. 1947

Surface Reflectance of Fibers

Synthetic fibers. Anon. *Dyer* **96**, 273–5 (Sept. 13, 1946).

A practical discussion is given of the physical properties which affect the surface reflectance and color of fibers and fabrics. J. A. Woodruff

Text. Research J. Feb. 1947

Properties of Regenerated Fibers

Regenerated fibers from natural polymers. D. Entwistle. *J. Soc. Dyers and Colourists* **62**, 262–71 (Sept. 1946).

The third John Mercer Lecture. A detailed discussion of the behavior of fibers and high polymeric systems generally in the light of today's knowledge, and an examination of the broad relationships between their raw materials, method of manufacture, fine structure, and physical properties. A comprehensive summary of the present status in this field, excellently organized. It is suggested that the limit in dry tenacity of the more extensible regenerated celluloses and cellulose acetate fibers has probably been approached, but that advances in the direction of decreased swelling and loss of tenacity when wet, improved torsional rigidity, and shear strength are possible. The functional character to be expected of textile materials of the future is emphasized. K. S. Campbell

Text. Research J. Feb. 1947

Water-Resistance of Fibers

Relation between water-resistance and water-repellency of viscose fibers. E. Lev and Z. Rogovin. *Tekstil. Prom.* **5**, No. 11/12, 45–6 (1945) (through *Chem. Abstr.* **40**, 5570[6] (Sept. 20, 1946)).

Viscose fabrics treated with (*a*) urea-CH_2O resin in combination with Al salts, (*b*) 4% solution of polyvinyl acetate, (*c*) 4% solution of polymethyl methacrylate, and (*d*) solution of Velan were submerged in H_2O and the time required for maximum elongation was determined. The treated fabrics required 6–10 times as long as the untreated. The elongation of the variously treated fabrics at equilibrium was practically the same. No relation was found between water-repellency and water-resistance (retention of strength when wet).

Text. Research J. Feb. 1947

Frictional Properties of Wool

Frictional properties of wool. Helen M. S. Thompson and J. B. Speakman. *Nature* **157**, 804 (June 1946).

Fibers of wool were coated with thin films of silver and gold and their frictional properties studied. Scaliness measurements by the violin-bow and lepidometer methods, as

well as the results of photomicrographic studies, are reported. From results on fibers with a coating of silver 0.03μ thick it is concluded that the frictional properties of wool are due to the ratchet effect of the scales. R. W. Eyler

Text. Research J. Feb. 1947

Static in Textile Processing

Static electricity in textile processing. Anon. *Silk J. Rayon World* 21, 34–6, 39 (Jan. 1946).

Troubles due to static have been aggravated by the introduction of manufactured fibers having low moisture regains and by the general tendency to increase processing speeds. Methods for reducing static effects are reviewed.

R. K. Worner

Text. Research J. Feb. 1947

Warping

Tricot requires careful warping. O. B. Graham. *Textile World* **96**, 116, 117, 204, 206 (Oct. 1946).

A few practical suggestions on the careful preparation of warps for tricot knitting. H. J. Burnham

Text. Research J. Feb. 1947

O.T.S. REPORTS*

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Dyes

Neue Farbstoffe und neue Färbereihilfsprodukte ab 1936, Werte aus 1941. (New dyes and dye auxiliaries, from 1936 to 1941; values as of 1941.) (FIAT Microfilm Reel C 25.) I. G. Farbenindustrie, A.G. PB 4683, Nov. 1945; 180 pp.; microfilm, $2.75—enlargement prints, $18.00 (through *Bib. Sci. and Ind. Reports* **3**, 313 (Nov. 1, 1946)).

This microfilm reel contains a tabulation of statistical information compiled by the "TEA-Büro" (Technical Committee) of the I. G. Farbenindustrie, A.G., pertaining to dyes and auxiliary compounds used in dyeing processes, which were developed and marketed during the years 1936 to 1941, inclusive. The first part of the document lists the total figures for 1941, giving the number of new products marketed, cost price, selling price, tonnage sold, and the proceeds therefrom. Subtotals are given for the various classes of products, such as: azo dyes, naphthol dyes, alizarin and vat dyes, substantive, indigo, sulfur dyes, those of the triphenylmethane and phthalocyanine groups, fluorene derivatives, and dyes for leather and fur. The second part lists the individual compounds (arranged by groups), and contains the following information: manufacturing plant, the inventor, the year in which the product was put on the market, the individual tonnage figures for the years 1936 to 1941, together with cost and selling price, a suggested standard price, and the income for the year 1941. The various textile aids and related compounds, for which no subtotals are given in part one, appear here individually, together with the dyes, also listed individually. A total of more than 1,000 compounds is tabulated. In German.

Text. Research J. Feb. 1947

Jute Manufacture

German jute industry. D. T. D. Bruce, and others. (BIOS Final Rept. 624, Item 22, 31) PB 48443, May-June, 1946; 17 pp.; microfilm, $1.00—photostat, $2.00 (through *Bib. Sci. and Ind. Reports* **3**, 802 (Dec. 13, 1946)).

Ten firms inspected are listed in Appendix A. Recommended as being suitable for the British trade are an individual bag cutter (Appendix C) and the Martin automatic cop loader (Appendix E) which are described in some detail. Also described are a device to increase doffing speed of old-type spinning frames (Appendix D) and a zell jute and tow card assembly. A small supply of raw jute is being manufactured into bags for use in Germany but the industry is still operating at a low level of efficiency and capacity according to the report. This is due to the shortage of coal, trained operatives, and raw materials such as zell jute, paper, and green flax. It is stated that the difficulty in weaving cops of rayon or fiber spun on jute machinery has been overcome in Germany by using zell jute, a rayon material. A method of applying one of the synthetic rubber materials to jute cloth was observed at Vereinigte Jute Spinnereien und Webereien.

Text. Research J. Feb. 1947

Patent Applications

Patent applications of the Photo and Artificial Silk Patent Section, Wolfen, 1939–1940, complete with index compiled by the Höchst patent office. (FIAT Microfilm Reel Patents 141.) I. G. Farbenindustrie, A.G., Wolfen. PB 19925, Mar. 1946; frames Nos. 1–1058, $6.00 (through *Bib. Sci. and Ind. Reports* **3**, 211 (Oct. 18, 1946)).

This is the first of 6 microfilm reels, the total containing the patent applications of the I.G. plant at Wolfen, from the second half of 1939 through 1945; reel 141 contains applications made in 1939 and 1940. A great variety of fields is covered, among them: color and black-and-white photographic emulsions, cameras, projectors and other photographic equipment; raw products and materials, methods and devices pertaining to rayon and other artificial fibers; plastics, particularly polyamides, their synthesis and conversion into useful forms; solvents, dyes and resins. These applications are available individually; short abstracts and the PB numbers under which these documents may be ordered appear on pages 212–24 of *Bibliography* **3** (Oct. 18, 1946).

Text. Research J. Feb. 1947

Textiles and Textile Products

Civil affairs information guide: The textile industry in France. (War Dept. Pamphlet 31-172.) U.S. Foreign Economic Administration. PB 43113, July 1944; 86

*Copies of these reports may be obtained from the Office of Technical Services, Department of Commerce, Washington 25, D. C. Orders should be accompanied by check or money order payable to the Treasurer of the United States.

pp.; microfilm, $2.00—photostat, $6.00 (through *Bib. Sci. and Ind. Reports* **3**, 589 (Nov. 22, 1946)).

This report, written when France was still occupied, analyzes the pre-war characteristics of the French textile industry and its branches. Against this background, it traces the developments from Sept., 1939, to the end of 1943, and then attempts to appraise the situation and problems likely to be found after liberation. Dependent on imports for more than 90% of its raw materials before the war, the industry deteriorated greatly during the occupation. It was reorganized along Fascist lines and produced mainly military clothing and other goods ordered by the Nazis. Early rehabilitation depends on the availability of power, transportation, fuel, and chemicals, and on the survival of manufacturing equipment. An appendix contains sources and statistical tables of production and organization.

Text. Research J. Feb. 1947

MISCELLANEOUS

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Conversion of Cellulose into Glucides

Cellulose and its conversion into glucides. Georges Champetier. *Chimie & industrie* **54**, 6–16 (1945) (through *Chem. Abstr.* **40**, 6810[5] (Nov. 10, 1946)).

An address reviewing, with 53 references, the various methods of converting cellulose into glucides, the characteristics of the products obtained, and the more important technical applications.

Text. Research J. Feb. 1947

Cellulose By-Products

The problem of substances which accompany cellulose destined for the production of textile fibers. Juan B. Puig. *Afinidad* **23**, 305–20 (1946) (through *Chem. Abstr.* **40**, 7614[9] (Nov. 20, 1946)).

A discursive review of the plant-physiological origins and chemistry of the various types of cellulose. An outstanding problem is the industrial utilization of the hemicelluloses, lignin, and the pectins.

Text. Research J. Feb. 1947

Cellulose Ethanol

Cellulose ethanol. Technique of hydrolysis, yields, installation costs. Ch. Berthelot. *Energie & mat. rempl.* **9**, No. 29, 10–11; No. 30, 8; No. 31, 3–5 (1941); *Chimie & industrie* **47**, 106 (1942) (through *Chem. Abstr.* **40**, 6745[5] (Nov. 10, 1946)).

Text. Research J. Feb. 1947

Handling Corrosive Liquids

Corrosive reagents in rayon manufacture. A. G. Arend. *Silk J. Rayon World* **23**, 38–9, 42 (Aug. 1946).

Improved equipment for storing and handling corrosive liquids in rayon manufacture is described.

Text. Research J. Feb. 1947 R. K. Worner

Retting of Jute

Retting by Hiparol and by bacteria. Parukutty Baruah and H. K. Baruah. *Science and Culture* **11**, 369–73 (1946) (through *Chem. Abstr.* **40**, 7645[4] (Nov. 20, 1946)).

Hiparol, a mixture of enzymes secreted by *Thielaviopsis paradoxa*, appeared to be more active than bacterial enzymes in the retting of jute and coconut husk fibers. The development of processes for recovering galacturonic acid, AcOH, butyric acid, Me_2CO, BuOH, and EtOH during hydrolysis by Hiparol, and bacterial enzymes is discussed.

Text. Research J. Feb. 1947

Substitute for Gum Tragacanth

"Gum taminda," a new substitute for gum tragacanth or carob bean gum. T. P. Ghose and S. Krishna. *Indian Text. J.* **66**, 506–8, 561 (Mar. 1946).

The preparation of tamarind-seed pectin and the reaction of borax with it to form "gum taminda" are described. This gum was developed during war shortages and has been used with success in cotton and jute mills as a substitute for gum tragacanth or carob bean.

Text. Research J. Feb. 1947 R. K. Worner

Artificial Leather

Manufacture of leather cloth in India. D. S. Pethe. *Indian Text. J.* **66**, 543, 555 (Mar. 1946).

A modification of nitrocellulose finish on cotton base fabric is used. Details of processing are described.

Text. Research J. Feb. 1947 R. K. Worner

Lithium Compounds

Organo-lithium compounds. Zeno W. Wicks. *Interchem. Rev.* **5**, 69–73 (Autumn, 1946).

This brief review is the first to appear in English on the preparation and reactions of compounds containing lithium bonded directly to carbon. Two methods of preparation and numerous examples of organic syntheses possible with organo-lithium compounds are cited, with emphasis on contrasts in behavior as compared to the analogous Grignard reagents. 25 references.

E. N. Harvey, Jr.

Text. Research J. Feb. 1947

Lubrication of Machinery

Lubricants and lubrication with special reference to weaving machinery. V. A. Ajgaonkar. *Indian Text. J.* **66**, 639–46 (Apr. 1946).

The importance of proper lubricants and adequate lubrication is emphasized. How the lubricant acts, various types of lubricants, properties and testing of lubricating oils, types of lubricants suitable for different machines, quantity of oil required for different machines, and application of the oil are discussed.

Text. Research J. Feb. 1947 R. K. Worner

Metal Powders

Metal powders. David B. Pall. *Interchem. Rev.* **5**, 59–68 (Autumn, 1946).

Various aspects of the manufacture and more recent applications of metal powders in powder metallurgy are reviewed and advantages of a

new molybdenum permalloy powder developed for use in electronic cores are discussed. E. N. Harvey, Jr.
Text. Research J. Feb. 1947

Casein Plastics

Protein plastics and fibres. Part I. S. H. Pinner. *Brit. Plastics* **18**, 313–8 (July 1946).

In this review the preparation and structure of casein plastics are discussed as well as the reaction with formaldehyde, water sorption, rheology, and improvements that have been made. E. F. Evans
Text. Research J. Feb. 1947

Protein Fibers

Protein plastics and fibers. Part II. S. H. Pinner. *Brit. Plastics* **18**, 353–61 (Aug. 1946).

A review of casein, soybean, zein, peanut, and egg albumin proteins and their use as fibers. 98 references. E. F. Evans
Text. Research J. Feb. 1947

German Cellulosic Plastics

The cellulose plastic industry in Germany. Extracts from a report by the British Intelligence Objectives Subcommittee. *Silk J. Rayon World* **22**, 39–40, 42, 55 (Apr. 1946).

This article contains extracts from the report of investigations, carried out by Edward C. May and Norman A. C. Friend of various firms engaged in the manufacture of cellulose plastics. R. K. Worner
Text. Research J. Feb. 1947

Thermoplastic Spinnerets

Spinnerets of thermo-plastic material. W. Jacobsohn. *Silk J. Rayon World* **23**, 52 (Aug. 1946).

Patent references and details of construction are given.
Text. Research J. Feb. 1947 R. K. Worner

Polysaccharides

The structure, function, and synthesis of polysaccharides. W. N. Haworth. *Proc. Roy. Soc.* **A186**, 1–19 (June 4, 1946).

This Bakerian Lecture is a highly condensed review which covers cellulose, starch, some of the dextrins, agar, and many polysaccharides of physiological and bacteriological interest. W. E. Davis
Text. Research J. Feb. 1947

British Patent Review

Progress in rayons and rayon processing. Anon. *Silk and Rayon* **20**, 1229–32 (Oct. 1946).

A brief discussion is given of the following subjects: (1) wet spinning of acetate rayon (B.P. 575,661); (2) lubricant for acetate yarns (B.P. 571,490); (3) detergent and emulsifying agents (B.P. 573,524); (4) suction for removing water (B.P. 571,575); (5) new azo dyes for nylon (B.P. 569,080); (6) antislip finishing agents (B.P. 574,644); (7) pile-brushing machine (B.P. 575,579).
Text. Research J. Feb. 1947 J. F. Keating

Urea derivatives instead of urea for textile treatment. Anon. *Silk and Rayon* **20**, 1234–8 (Oct. 1946).

A brief discussion is given of the following subjects: (1) retention, after dyeing, of low luster in delustered acetate rayon by the use of melamine-formaldehyde resins (B.P. 568,438); (2) inhibiting gas fading by the use of melamine-formaldehyde resins (B.P. 569,577); (3) water-in-oil types of printing pastes containing a mixture of urea and melamine resins (B.P. 573,558); (4) pigment binding agent consisting of modified melamine resins (B.P. 538,993, B.P. 539,737, and B.P. 573,368); (5) improvement of color fastness with melamine-formaldehyde resins (B.P. 482,345 and B.P. 532,871); (6) flame-resistance by use of a melamine-formaldehyde resin plasticized with a chlorinated alkyl ester of phosphoric acid (B.P. 564,573). J. F. Keating
Text. Research J. Feb. 1947

Pectinolytic Organisms

Pectinolytic properties and retting by *Clostridium corallinum* P. and R. André Romain Prévot and Marcel Raynaud. *Compt. rend.* **222**, 1531–3 (1946) (through *Chem. Abstr.* **40**, 6538[7] (Nov. 10, 1946)).

A biochemical study of *Cl. corallinum* is presented in which its pectinolytic properties are compared with those of other similar species and its value as a retting organism for flax, hemp, ramie, broom, and alfa is emphasized. It is shown to be different from *Cl. felsineum*, *Cl. roseum*, and *Cl. haumanni*, with which it forms a very homogeneous natural group of chromogenic, pectinolytic, anaerobic organisms.
Text. Research J. Feb. 1947

Polymer Research

Recent progress in polymer research. H. Mark. *Record Chem. Progress* **7**, 27–31 (1946) (through *Chem. Abstr.* **40**, 6880[2] (Nov. 10, 1946)).

A review with 116 references.
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Alginate Rayon

Some uses of calcium alginate rayon. A. Johnson and J. B. Speakman. *J. Soc. Dyers and Colourists* **62**, 97–100 (Apr. 1946).

Calcium alginate rayon, which is readily spun, possesses satisfactory elastic properties for weaving and knitting, but is dissolved by dilute solutions of alkali. After a number of alkali-resistant alginate rayons had been developed, attention was turned to the possible use of calcium alginate rayon to produce special types of fabric by taking advantage of its defect. With the help of the rayon, simplified methods of making crepes, astrakhans, and pile fabrics have been evolved, and new methods of ornamenting textile materials developed. In addition, a general method of making fabrics composed of entirely twistless yarns has been discovered. The special merit of these processes is that they can be carried out with all ordinary types of textile fiber, without introducing an extra process into the finishing routine, because calcium alginate rayon can be removed from fabrics by a normal scouring process.
Text. Research J. Feb. 1947 Authors

German Rayon Objectives

A survey of the German rayon staple industry. Extracts from British Intelligence Objectives Subcommittee Final Report No. 50. *Silk J. Rayon World* **23**, 32–4, 47 (Aug. 1946).

Economic and political forces affecting the rayon industry are discussed. The industry was divided into four combines or rings which included the following objectives in their economic plan: substitution of German beech, pine, and straw pulps for Scandinavian spruce; reduction of chemical consumption and recovery of chemical waste; development of continuous alkali cellulose processes to conserve iron and manpower; development of various methods and treatments to increase wear-resistance and laundering-resistance of rayon; development of cellulose fibers with water-resistance and other wool-like properties; development of specialty cellulose fibers, such as jute type, etc.; and development of fully synthetic staple fibers to fulfill specific requirements. R. K. Worner

Text. Research J. Feb. 1947

Acenaphthylene Resins

New plastics from coal-tar. J. Idris Jones. *Brit. Plastics* **18**, 286–9 (July 1946).

Acenaphthene found in coal-tar is dehydrogenated to yield acenaphthylene. The latter can be polymerized in bulk by heating at 98°C for 72 hrs. to yield a resin of molecular weight of 90,000 which is soluble in benzene, or by heating with benzoyl peroxide to a high-molecular-weight polymer. It can also be polymerized in solution and at low temperatures with ionic-type catalysts. Acenaphthylene will copolymerize with other monomers such as styrene, methyl methacrylate, and vinyl carbazole. The polymers are useful as film-forming materials, as coating agents, as bonding agents, as impregnants, as molding materials, and particularly as dielectric materials. E. F. Evans

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Natural History of Silk

The natural history of silk. Eric Hardy. *Indian Text. J.* **66**, 441–3 (Feb. 1946).

A review. R. K. Worner

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Disinfection of Wool

Use of chloramine with ammonia as activator for disinfecting of wool infected with spores of anthrax. R. M. Ginsburg and P. F. Milyavskaya. *Hig. i Sanit.* **1945**, No. 1/2, 45 (through *Chem. Abstr.* **40**, 7645[8] (Nov. 20, 1946)).

Solutions of chloramine in 2–3% concentration plus small quantities of NH_3 disinfected previously washed woolen goods in 30 min. Unwashed wool was disinfected by a concentration of 0.3–1.0% of chloramine in presence of NH_3. The color of wool was slightly dulled by the treatment, but other properties remained unchanged.

Text. Research J. Feb. 1947

Pneumatic Shuttle Propulsion

Electro-pneumatic shuttle propulsion. D. C. Snowden. *Silk J. Rayon World* **23**, 28–31 (Aug. 1946).

This development by the Electro-Pneumatic Looms Ltd., Bradford, England, features the propelling of the shuttle across the race by released compressed air. It makes possible greatly increased loom speeds, resulting in increased production. R. K. Worner

Text. Research J. Feb. 1947

Research Library

What a research library offers. Lisa G. Otto. *Interchem. Rev.* **5**, 42–6 (Summer, 1946).

The various services offered by an industrial research library are reviewed. E. N. Harvey, Jr.

Text. Research J. Feb. 1947

Textile Research

Concerning textile research. Edward R. Schwarz. *Am. Dyestuff Reptr.* **35**, 107–10 (Feb. 25, 1946).

The premise is offered that the textile field can be divided into two branches of study, the "inherent properties of textile materials" and the "form in which the material is to be found." J. A. Woodruff

Text. Research J. Feb. 1947

Technical Education

Education and the bleaching, dyeing, printing, and finishing industries. A. W. Doyle. *J. Soc. Dyers and Colourists* **62**, 101–8 (Apr. 1946).

The history of technical education in England is given, followed by suggested plans for increased development of technical training for those who cannot afford to pay for their education. Industry is assumed to have a large responsibility in making possible such education in order to increase its own efficiency. The practicability of part-time day schooling is discussed. 21 references. J. A. Woodruff

Text. Research J. Feb. 1947

Comparison of British, American, and Indian Textile Industries

Wartime developments and trends in textile industry. Lalbhai R. Mehta. *Indian Text. J.* **66**, 723–9 (May 1946).

The textile industries of the United States, United Kingdom, and India are compared with special reference to the cotton textile industry.

Text. Research J. Feb. 1947 R. K. Worner

Textile Map of India

The textile map of India. Indian Textile Engineers, Ltd. *Indian Text. J.* **66**, 612 (Supplement) (Apr. 1946).

A map of India is presented. It shows every town in which cotton mills are situated, with the number of spindles and looms installed on Jan. 1, 1946; also the position of all high-tension electric grids from which power can be obtained for industrial and other uses.

Text. Research J. Feb. 1947 R. K. Worner